Supporting Information

α,β-Divinyl Tetrahydropyrroles as Chiral Chain Diene Ligands in Rhodium(I)-Catalyzed Enantioselective Conjugated Additions

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Contents

1. General

Air and moisture sensitive reactions were carried out in oven-dried glassware sealed with rubber septa under a positive pressure of dry argon. Similarly sensitive liquids and solutions were transferred via syringe. Reactions were stirred using Teflon-coated magnetic stir bars. Elevated temperatures were maintained using Thermostat-controlled silicone oil baths. Organic solutions were concentrated using a Büchi rotary evaporator with a desktop vacuum pump. Dioxane and synthetic reagents were purchased from Acros, Aldrich, and Alfa Aesar and used without further purification, unless otherwise indicated. Analytical TLC was performed with 0.25 mm silica gel G plates with a 254 nm fluorescent indicator. The TLC plates were visualized by ultraviolet light and treatment with phosphomolybdic acid stain followed by gentle heating. Purification of products was accomplished by flash chromatography on silica gel and the purified compounds show a single spot by analytical TLC.

NMR spectra were measured on Bruker ARX 400 (^{1}H at 400 MHz, ^{13}C at 100 MHz) nuclear magnetic resonance spectrometers. Data for ^{1}H-NMR spectra are reported as follows: chemical shift (ppm, referenced to TMS; s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, dt = doublet of triplets, ddd = doublet of doublet of doublets, ddt = doublet of doublet of triplets, m = multiplet), coupling constant (Hz), and integration. Data for ^{13}C-NMR are reported in terms of chemical shift (ppm) relative to residual solvent peak (CDCl$_3$: 77.0 ppm). Infrared spectra were recorded on an AVATAR 330 Fourier transform spectrometer (FT-IR) with an OMNI sampler and are reported in wavenumbers (cm^{-1}). High-resolution mass spectra (HRMS) were recorded on a Bruker Apex IV FTMS mass spectrometer (ESI). Optical rotations were measured on a Perkin-Elmer 341 LC spectrometer. The enatiomeric excesses (ee) of the products were determined by chiral HPLC analysis using Aglient HP 1100 instrument.

Abbreviations:

coe = (Z)-cyclooctene

2. Representative Procedures for the Rh(I) catalyzed Conjugated Addition of Phenyl Boronic Acids to 2-cyclohexenone

To a Schlenk flask charged with phenyl boronic acid (60.0 mg, 0.49 mmol), [Rh(coe)$_2$Cl]$_2$ (6.0 mg, 8.36 μmol, 2.5 mol %), and chiral diene ligand **1a** (94% ee, 7.0 mg, 20 μmol, 6.0 mol %) was added degassed dioxane (0.9 mL) under Argon. The resulting mixture was heated to 50 ℃ and stirred for 15 min, followed by addition of 2-cyclohexenone (32.1 mg, 0.33 mmol) and aq. KOH (0.033 mmol, 0.075 M, 0.45 mL). The reaction mixture was stirred at room temperature for 2 h, solvent was removed under reduced pressure. The crude residue was purified by flash chromatography on silica gel (hexanes: ethyl acetate = 30:1, v/v) to give the conjugated addition product as a colorless oil (51.3 mg, 89% yield, 91% ee).
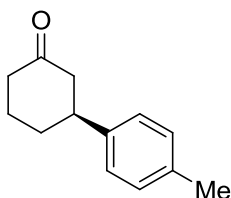
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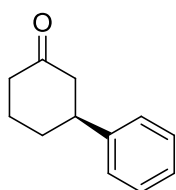
Colorless oil, $[\alpha]_D^{20}$ = -18.9 (*c* 1.33, CDCl$_3$) (91% ee) [lit.: $[\alpha]_D^{23}$ = -19.5 (*c* 0.95, CHCl₃) (93% ee) for *S*-isomer]. ^{1}H-NMR (400 MHz, CDCl$_3$): δ 7.36-7.30 (m, 2H), 7.27-7.19 (m, 3H), 3.06-2.96 (m, 1H), 2.64-2.32 (m, 4H), 2.19-2.05 (m, 2H), 1.92-1.71 (m, 2H).
C.-G., Feng, Z.-Q. Wang, C. Shao, M.-H. Xu, G.-Q. Lin, *Org. Lett.* **2008**, *10*, 4101.
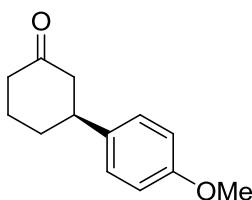
Table 2, entry 2

Colorless oil, $[\alpha]_D^{20}$ = -17.1 (*c* 1.29, CDCl$_3$) (88% ee) [lit.: $[\alpha]_D^{23}$ = -15.0 (*c* 1.09, CHCl₃) (94% ee) for *S*-isomer]. ^{1}H-NMR (400 MHz, CDCl$_3$): δ 7.16-7.09 (m, 4H), 3.02-2.93 (m, 1H), 2.61-2.35 (m, 4H), 2.33 (s, 3H), 2.18-2.02 (m, 2H), 1.89-1.70 (m, 2H).
C.-G., Feng, Z.-Q. Wang, C. Shao, M.-H. Xu, G.-Q. Lin, *Org. Lett.* **2008**, *10*, 4101.

Table 2, entry 3

Colorless oil, $[\alpha]_D^{20}$ -18.3 (*c* 0.8, CDCl$_3$) (89% ee) [lit.: $[\alpha]_D^{23}$ -14.2 (*c* 1.02, CHCl$_3$) (92% ee) for *S*-isomer].
^{1}H-NMR (400 MHz, CDCl$_3$): δ 7.14 (dt, *J* = 8.4 and 2.0 Hz, 2H), 6.87 (dt, *J* = 8.4 and 2.0 Hz, 2H), 3.79 (s, 3H), 3.01-2.91 (m, 1H), 2.60-2.31 (m, 4H), 2.17-2.02 (m, 2H), 1.87-1.69 (m, 2H).
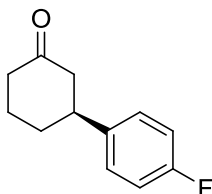
C.-G., Feng, Z.-Q. Wang, C. Shao, M.-H. Xu, G.-Q. Lin, *Org. Lett.* **2008**, *10*, 4101.
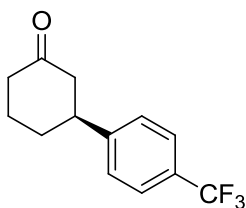
Table 2, entry 4

Colorless oil, $[\alpha]_D^{20}$ -15.3 (*c* 1.18, CDCl$_3$) (90% ee) [lit.: $[\alpha]_D^{20}$ +14.3 (*c* 1.2, CHCl$_3$) (98% ee) for *R*-isomer].
^{1}H-NMR (400 MHz, CDCl$_3$): δ 7.21-7.15 (m, 2H), 7.05-6.98 (m, 2H), 3.05-2.95 (m, 1H), 2.62-2.32 (m, 4H), 2.19-2.11 (m, 1H), 2.11-2.03 (m, 1H), 1.88-1.70 (m 2H).

M. Pucheault, S. Darses, J. P. Genet, *Eur. J. Org. Chem.* **2002**, 3552.

Table 2, entry 5

Colorless oil, $[\alpha]_D^{20}$ -11.8 (*c* 1.12, CDCl$_3$) (91% ee) [lit.: $[\alpha]_D^{23}$ -11.4. (*c* 0.95, CHCl$_3$) (95% ee) for *S*-isomer].
^{1}H-NMR (400 MHz, CDCl$_3$): δ 7.59 (d, *J* = 8.4 Hz, 2H), 7.35 (d, *J* = 8.4 Hz, 2H), 3.14-3.04 (m, 1H), 2.64-2.35 (m, 4H), 2.22-2.13 (m, 1H), 2.13-2.06 (m, 1H), 1.94-1.73 (m, 2H).

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Table 2, entry 6
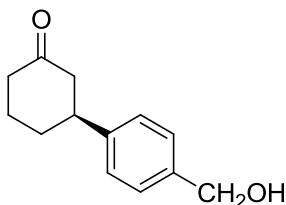
Corlorless oil, $[\alpha]_D^{20}$ -18.8 (*c* 0.97, CDCl$_3$) (92% ee). ^{1}H-NMR (400 MHz, CDCl$_3$): δ 7.32 (d, *J* = 8.2 Hz, 2H), 7.20 (d, *J* = 8.2 Hz, 2H), 4.65 (s, 2H), 3.04-2.95 (m, 1H), 2.59-2.23 (m, 5H), 2.18-2.02 (m, 2H), 1.89-1.70 (m, 2H). ^{13}C-NMR (100 MHz, CDCl$_3$): δ 211.2, 143.6, 139.3, 127.3, 126.6, 64.8, 48.8, 44.4, 41.1, 32.7, 25.4. IR

(neat): υ 3676-3091(br), 2942, 2875, 1713, 1523, 1453, 1427. HRMS (ESI) calcd for $C_{13}H_{16}NaO_2$ (M+Na)$^+$: 227.1043. Found: 227.1038.

Table 2, entry 7
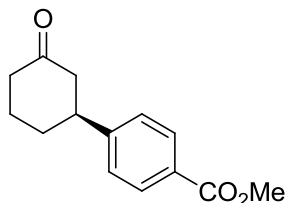
Corlorless oil, $[\alpha]_D^{20}$ -8.6 (*c* 1.22, CDCl$_3$) (87% ee). ^{1}H-NMR (400 MHz, CDCl$_3$): δ 8.00 (d, *J* = 8.3 Hz, 2H), 7.30 (d, *J* = 8.3 Hz, 2H), 3.91 (s, 3H), 3.12-3.03 (m, 1H), 2.64-2.35 (m, 4H), 2.21-2.06 (m, 2H), 1.93-1.73 (m, 2H). ^{13}C-NMR (100 MHz, CDCl$_3$): δ 210.2, 166.7, 149.4, 130.0, 128.6, 126.6, 52.0, 48.4, 44.6, 41.0, 32.4, 25.4. IR (neat): υ 3676-3091(br), 2965, 2935, 2875, 1724, 1616, 1438. HRMS (ESI) calcd for $C_{14}H_{16}NaO_3$ (M+Na)$^+$: 255.0992. Found: 255.0987.

Table 2, entry 8
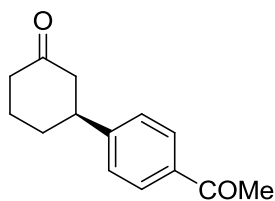
Colorless oil, $[\alpha]_D^{20}$ -8.7 (*c* 1.09, CDCl$_3$) (87% ee) [lit.: $[\alpha]_D^{32}$ -7.8 (*c* 1.02, CHCl$_3$) (95% ee) for *S*-isomer]. ^{1}H-NMR (400 MHz, CDCl$_3$): δ 7.94 (d, *J* = 8.0 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 3.14-3.04 (m, 1H), 2.60 (s, 3H), 2.59-2.36 (m, 4H), 2.22-2.14 (m, 1H), 2.14-2.06 (m, 1H), 1.95-1.76 (m, 2H).
C. Defieber, J.-F. Paquin, S. Serna, E. M. Carreira *Org. Lett.* **2004**, *6*, 3873-3876.

Table 2, entry 9

Colorless oil, $[\alpha]_D^{20}$ -18.4 (*c* 1.03, CDCl$_3$) (90% ee) [lit.: $[\alpha]_D^{25}$ -17.5 (*c* 1.02, CHCl$_3$) (94% ee) for *S*-isomer]. ^{1}H-NMR (400 MHz, CDCl$_3$): δ 7.25-7.18 (m, 1H), 7.07-6.98 (m, 3H), 3.01-2.91 (m, 1H), 2.61-2.32 (m, 4H), 2.34 (s, 3H), 2.19-2.10 (m, 1H), 2.10-2.02 (m, 1H), 1.90-1.70 (m, 2H).
C.-G., Feng, Z.-Q. Wang, C. Shao, M.-H. Xu, G.-Q. Lin, *Org. Lett.* **2008**, *10*, 4101.
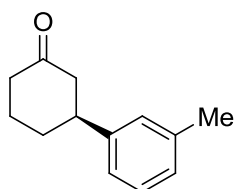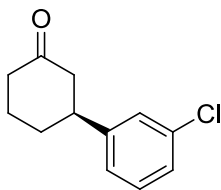
Table 2, entry 10

Colorless oil, $[\alpha]_D^{20}$ -10.9 (*c* 1.08, CDCl$_3$) (88% ee) [lit.: $[\alpha]_D^{25}$ -10.1 (*c* 1.05, CHCl$_3$) (94% ee) for *S*-isomer].
^{1}H-NMR (400 MHz, CDCl$_3$): δ 7.29-7.19 (m, 3H), 7.12-7.08 (m, 1H), 3.04-2.93 (m, 1H), 2.62-2.32 (m, 4H), 2.20-2.11 (m, 1H), 2.11-2.03 (m, 1H), 1.90-1.70 (m, 2H).
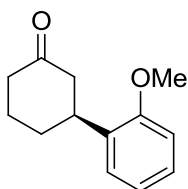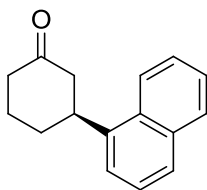C.-G., Feng, Z.-Q. Wang, C. Shao, M.-H. Xu, G.-Q. Lin, *Org. Lett.* **2008**, *10*, 4101.

Table 2, entry 11

Colorless oil, $[\alpha]_D^{20}$ -35.8 (*c* 1.43, CDCl$_3$) (89% ee) [lit.: $[\alpha]_D^{23}$ -26.5 (*c* 1.01, CHCl$_3$) (69% ee) for *S*-isomer].
^{1}H-NMR (400 MHz, CDCl$_3$): δ 7.24-7.17 (m, 2H), 6.94 (td, *J* = 7.3 and 1.0 Hz, 1H), 6.87 (dd, *J* = 8.5 and 1.0 Hz, 1H), 3.82 (s, 3H), 3.46-3.37 (m, 1H), 2.62-2.32 (m, 4H), 2.16-2.07 (m, 1H), 2.06-1.98 (m, 1H), 1.93-1.71 (m, 2H).
C.-G., Feng, Z.-Q. Wang, C. Shao, M.-H. Xu, G.-Q. Lin, *Org. Lett.* **2008**, *10*, 4101.

Table 2, entry 12

White solid, $[\alpha]_D^{20}$ -71.7 (*c* 1.02, CDCl$_3$) (88% ee) [lit.: $[\alpha]_D^{23}$ -31.7 (*c* 0.97, CHCl$_3$) (52% ee) for *S*-isomer].
^{1}H-NMR (400 MHz, CDCl$_3$): δ 8.03 (d, *J* = 8.4 Hz, 1H), 7.87 (d, *J* = 8.8 Hz, 1H), 7.75 (d, *J* = 8.4 Hz, 1H), 7.56-7.44 (m, 3H), 7.39 (d, *J* = 7.2 Hz, 1H), 3.91-3.80 (m, 1H), 2.81-2.39 (m, 4H), 2.29-2.13 (m, 2H), 2.06-1.85 (m, 2H).
C.-G., Feng, Z.-Q. Wang, C. Shao, M.-H. Xu, G.-Q. Lin, *Org. Lett.* **2008**, *10*, 4101.
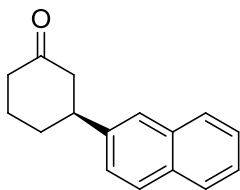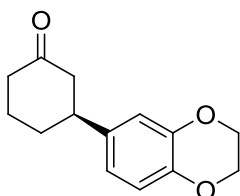
Table 2, entry 13

White solid, $[\alpha]_D^{20}$ -8.3 (*c* 1.42, CDCl$_3$) (88% ee) [lit.: $[\alpha]_D^{20}$ -8.3 (*c* 0.89, CHCl$_3$) (99% ee) for *S*-isomer].
^{1}H-NMR (400 MHz, CDCl$_3$): δ 7.83-7.76 (m, 3H), 7.62 (s, 1H), 7.49-7.40 (m, 2H), 7.34 (dd, *J* = 8.4 and 1.1 Hz, 1H), 3.20-3.09 (m, 1H), 2.71-2.56 (m, 2H), 2.51-2.32 (m, 2H), 2.20-2.08 (m, 2H), 1.98-1.72 (m, 2H).
Takaya, Y.; Ogasawara, M.; Hayashi, T. *Tetrahedron Lett.* **1999**, *40*, 6957.

Table 2, entry 14

Colorless oil, $[\alpha]_D^{20}$ -10.9 (*c* 1.05, CDCl$_3$) (91% ee). ^{1}H-NMR (400 MHz, CDCl$_3$): δ 6.81 (d, *J* = 8.4 Hz, 1H), 6.73 (d, *J* = 2.0 Hz, 1H), 6.68 (dd, *J* = 8.4 and 2.0 Hz, 1H), 4.24 (s, 4H), 2.95-2.83 (m, 1H), 2.59-2.29 (m, 4H), 2.17-1.99 (m, 2H), 1.84-1.67 (m, 2H), ^{13}C-NMR (100 MHz, CDCl$_3$): δ 211.0, 143.4, 142.1, 137.8, 119.4, 117.2, 115.1, 64.4, 64.3, 49.1, 44.0, 41.1 32.9, 25.4. IR (neat): υ 3676-3091(br), 2935, 2883, 1706, 1598, 1512, 1464, 1438. HRMS (ESI) calcd for C$_{14}$H$_{16}$NaO$_3$ (M+Na)$^+$: 255.0992. Found: 255.0984.

Table 2, entry 15
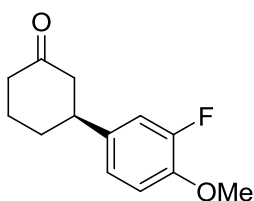
Colorless oil, $[\alpha]_D^{20}$ -15.5 (*c* 0.80, CDCl$_3$) (89% ee). ^{1}H-NMR (400 MHz, CDCl$_3$): δ 6.99-6.90 (m, 3H), 3.87 (s, 3H), 3.00-2.90 (m, 1H), 2.60-2.31 (m, 4H), 2.18-2.02 (m, 2H), 1.85-1.69 (m, 2H). ^{13}C-NMR (100 MHz, CDCl$_3$): δ 210.6, 153.5, 151.1, 146.2, 146.1, 137.4, 122.04, 122.01, 114.3, 114.1, 113.5, 56.23, 56.20, 48.9, 43.7, 41.0, 32.7, 25.3. The redundant peaks are for the splitting of fluorine atom. IR (neat): υ 3676-3091(br), 2965, 2935, 2879, 1717, 1631, 1594, 1523, 1468, 1453. HRMS (ESI) calcd for C$_{13}$H$_{15}$FNaO$_2$ (M+Na)$^+$: 245.0948. Found: 245.0943.

Table 2, entry 16
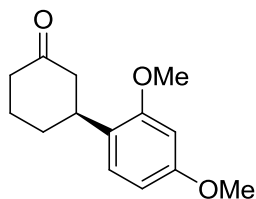
Colorless oil, $[\alpha]_D^{20}$ -25.4 (c 0.97, CDCl$_3$) (87% ee). ^{1}H-NMR (400 MHz, CDCl$_3$): δ 7.07 (d, J = 8.8 Hz, 1H), 6.48-6.44 (m, 2H), 3.789 (s, 3H), 3.786 (s, 3H), 3.37-3.26 (m, 1H), 2.58-2.30 (m, 4H), 2.14-2.05 (m, 1H), 2.03-1.95 (m, 1H), 1.89-1.69 (m, 2H). ^{13}C-NMR (100 MHz, CDCl$_3$): δ 211.6, 159.2, 157.5, 126.8, 125.0, 103.8, 98.5, 55.2, 55.1, 47.7, 41.2, 37.5, 31.1, 25.4. IR (neat): υ 3676-3091(br), 3013, 2950, 2872, 2846, 1713, 1620, 1594, 1512, 1468. HRMS (ESI) calcd for C$_{14}$H$_{18}$NaO$_3$ (M+Na)$^+$: 257.1148. Found: 257.1143.

Table 2, entry 17
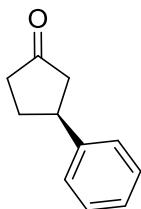
Colorless oil, $[\alpha]_D^{20}$ -82.0 (c 1.22, CDCl$_3$) (88% ee) [lit.: $[\alpha]_D^{24}$ -73.8 (c 1.21, CHCl$_3$) (80% ee) for S-isomer]. ^{1}H-NMR (400 MHz, CDCl$_3$): δ 7.37-7.31 (m, 2H), 7.28-7.22 (m, 3H), 3.48-3.60 (m, 1H), 2.71-2.62 (m, 1H), 2.51-2.40 (m, 2H), 2.39-2.24 (m, 2H), 2.05-1.93 (m, 1H).
C.-G., Feng, Z.-Q. Wang, C. Shao, M.-H. Xu, G.-Q. Lin, *Org. Lett.* **2008**, *10*, 4101.

Table 2, entry 18
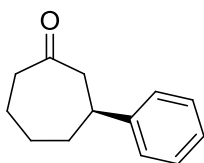
Colorless oil, $[\alpha]_D^{20}$ -53.3 (c 1.30, CDCl$_3$) (85% ee) [lit.: $[\alpha]_D^{33}$ +116.8 (c 0.35, CHCl$_3$) (95% ee) for R-isomer]. ^{1}H-NMR (400 MHz, CDCl$_3$): δ 7.32-7.24 (m, 2H), 7.22-7.14 (m, 3H), 2.98-2.84 (m, 2H), 2.69-2.52 (m, 3H), 2.12-1.94 (m, 3H), 1.82-1.64 (m, 2H), 1.55-1.42 (m, 1H).
C. Defieber, J.-F. Paquin, S. Serna, E. M. Carreira, *Org. Let.* **2004**, *6*, 3873.

Table 2, entry 19
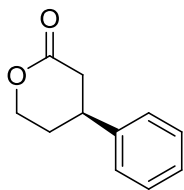
Colorless oil, $[\alpha]_D^{20}$ +2.7 (*c* 1.32, CDCl$_3$) (92% ee) [lit.: $[\alpha]_D^{25}$ +2.4 (*c* 1.03, CHCl$_3$) (80% ee) for *S*-isomer].
^{1}H-NMR (400 MHz, CDCl$_3$): δ 7.39-7.33 (m, 2H), 7.30-7.25 (m, 1H), 7.24-7.19 (m, 2H), 4.51 (ddd, *J* = 11.9, 4.9 and 4.0 Hz, 1H), 4.39 (ddd, *J* = 11.9, 10.3 and 4.0 Hz, 1H), 3.29-3.19 (m, 1H), 2.92 (ddd, *J* = 17.7, 5.9 and 1.5 Hz, 1H), 2.63 (dd, *J* = 17.7 and 10.3 Hz, 1H), 2.22-2.14 (m, 1H), 2.09-1.99 (m, 1H).
C.-G., Feng, Z.-Q. Wang, C. Shao, M.-H. Xu, G.-Q. Lin, *Org. Lett.* **2008**, *10*, 4101.

3.^{1}H and ^{13}C-NMR Spectra for New Compounds

Table 2, entry 1
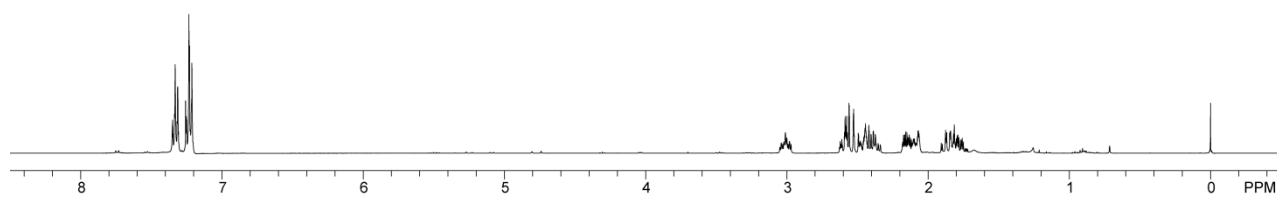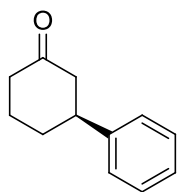
Table 2, entry 2
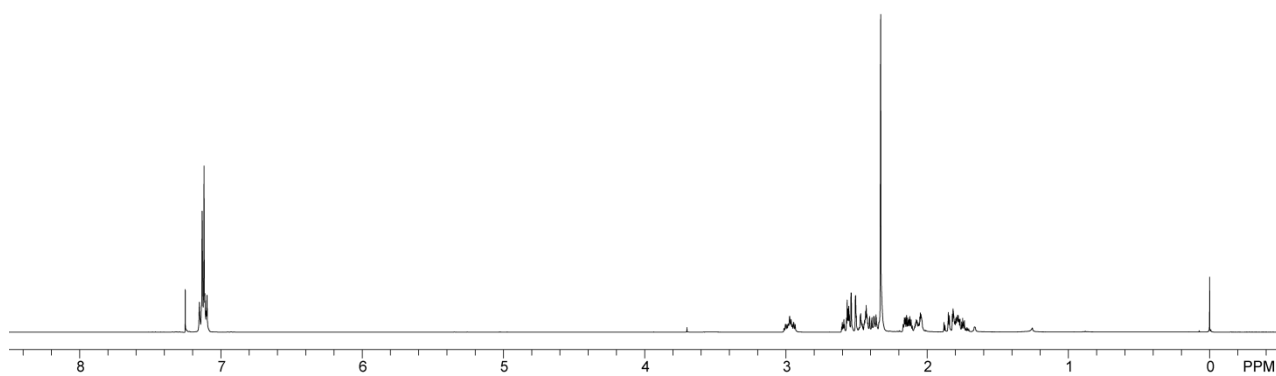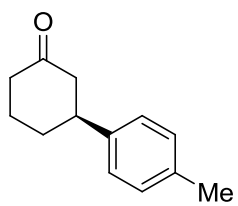
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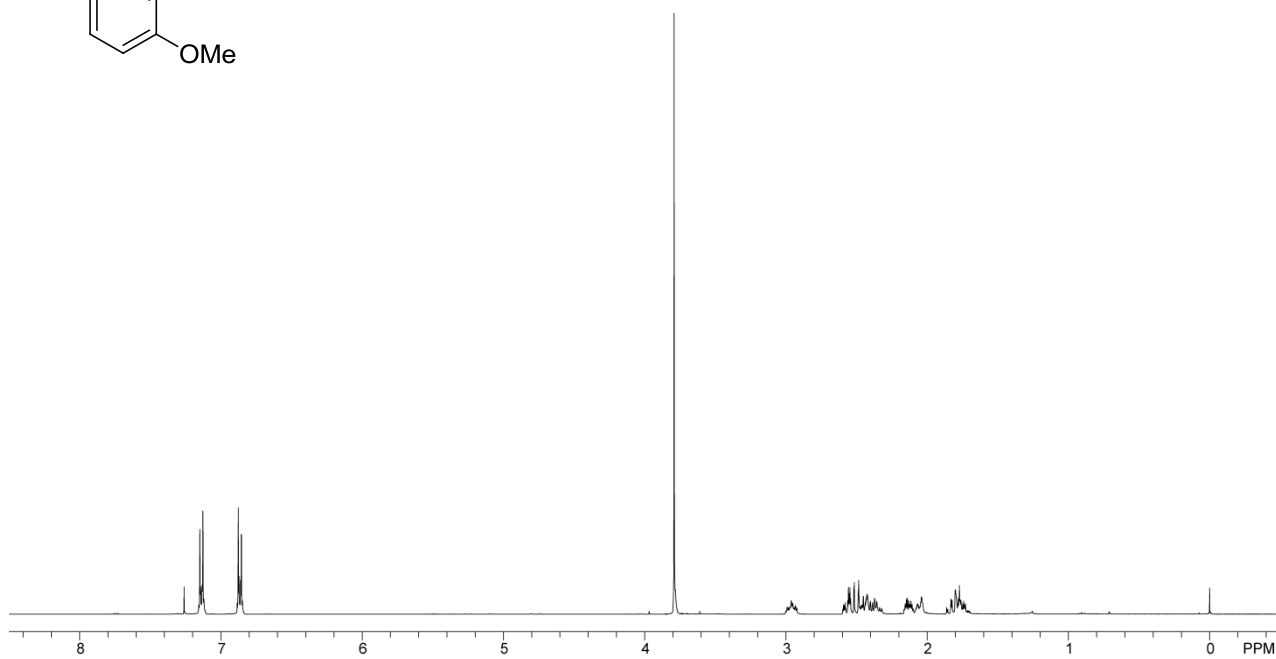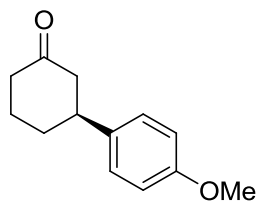
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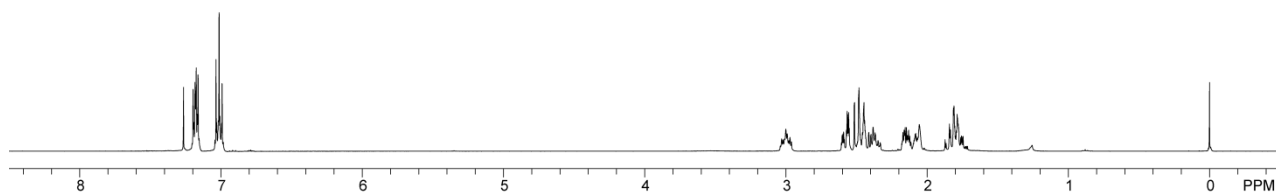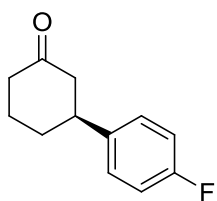
Table 2, entry 5
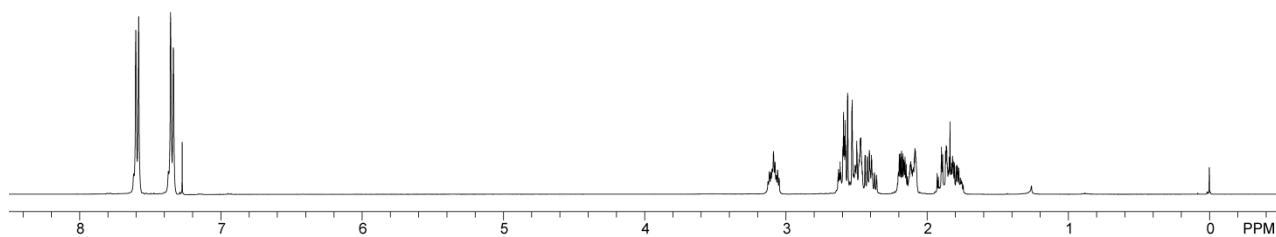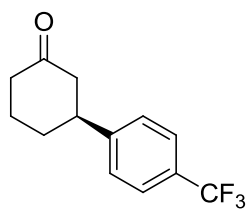
Table 2, entry 6
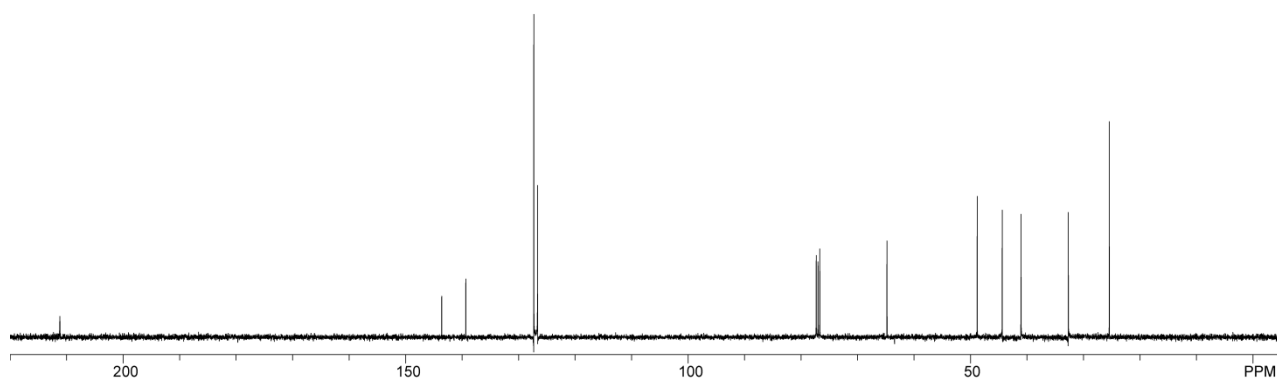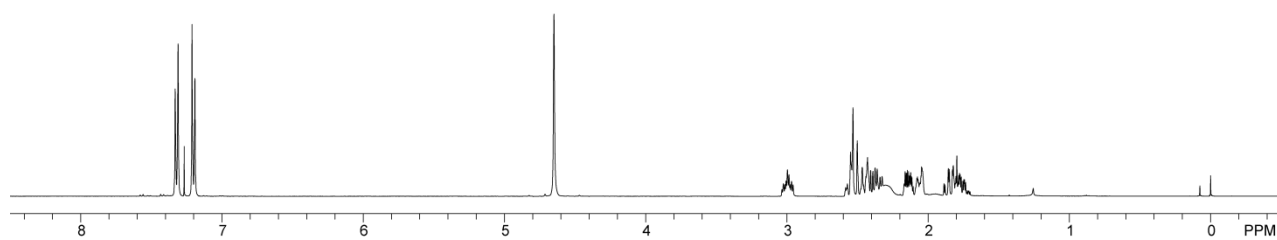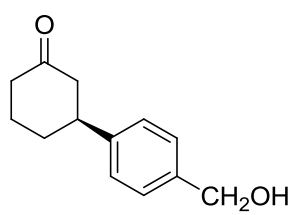
Table 2, entry 7
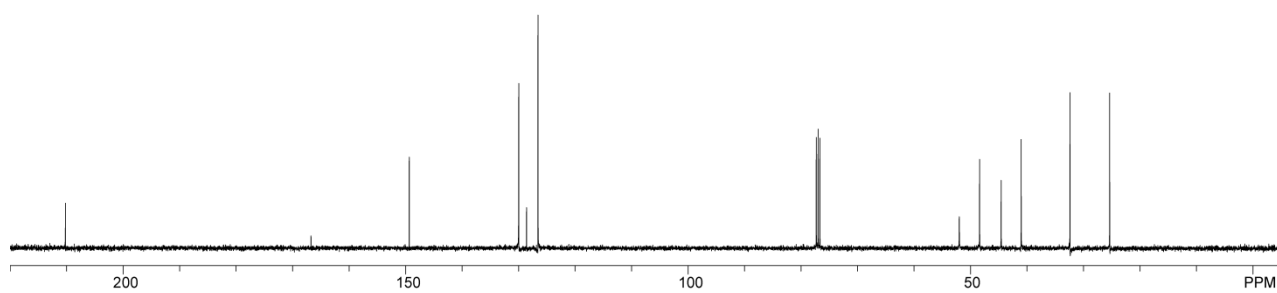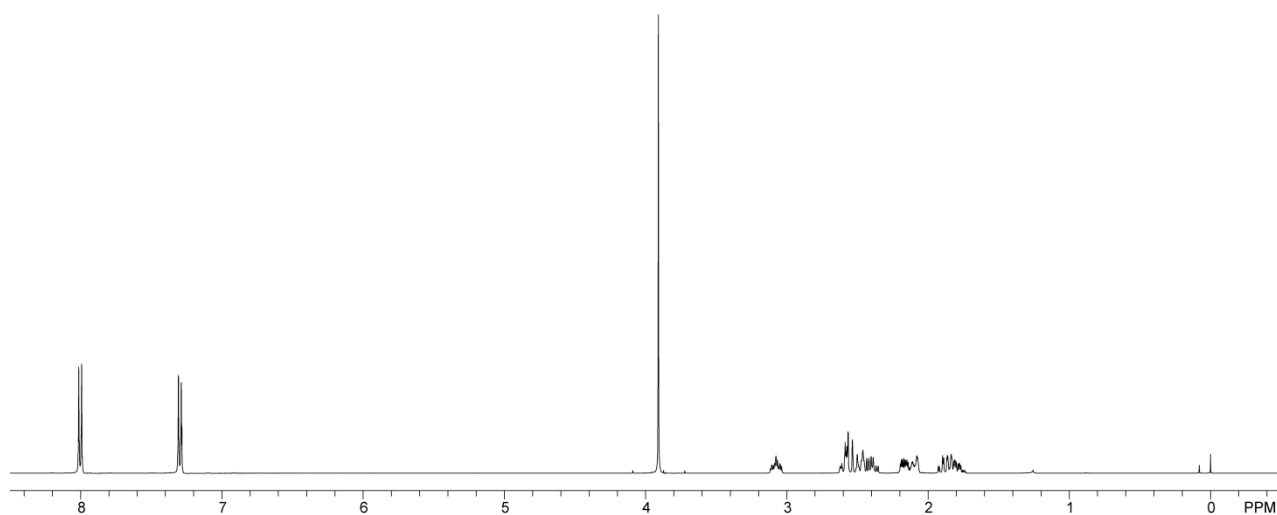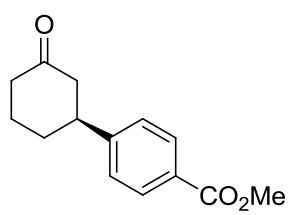
Table 2, entry 8
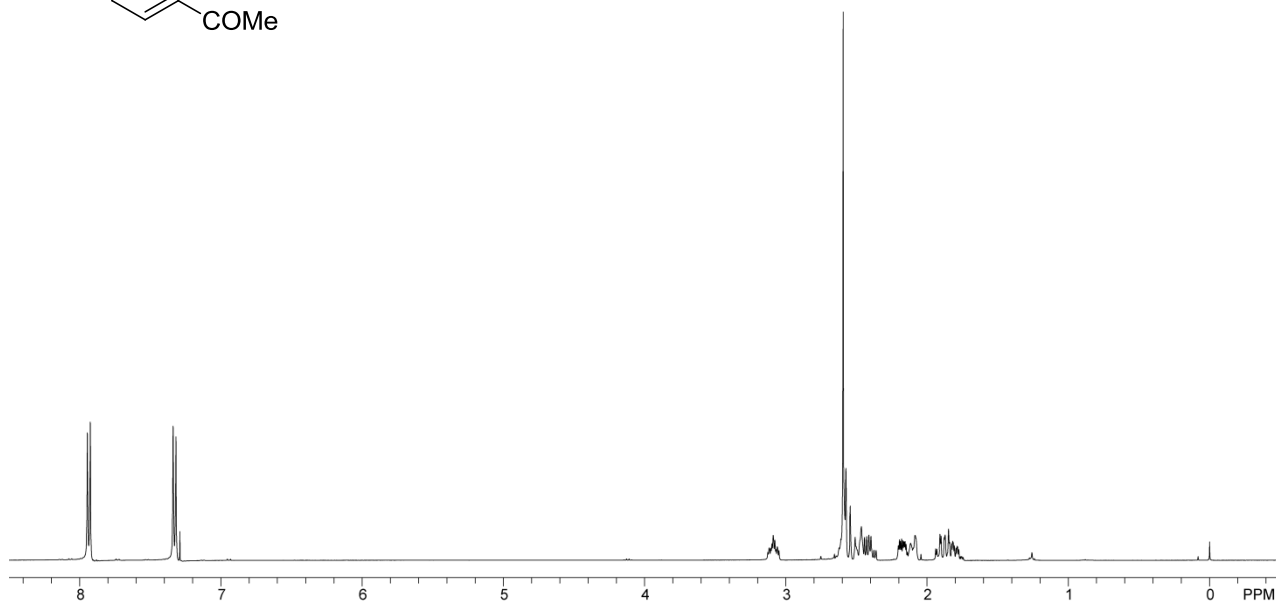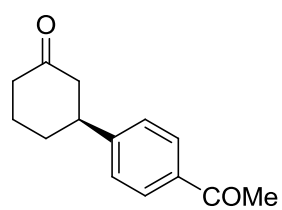
Table 2, entry 9
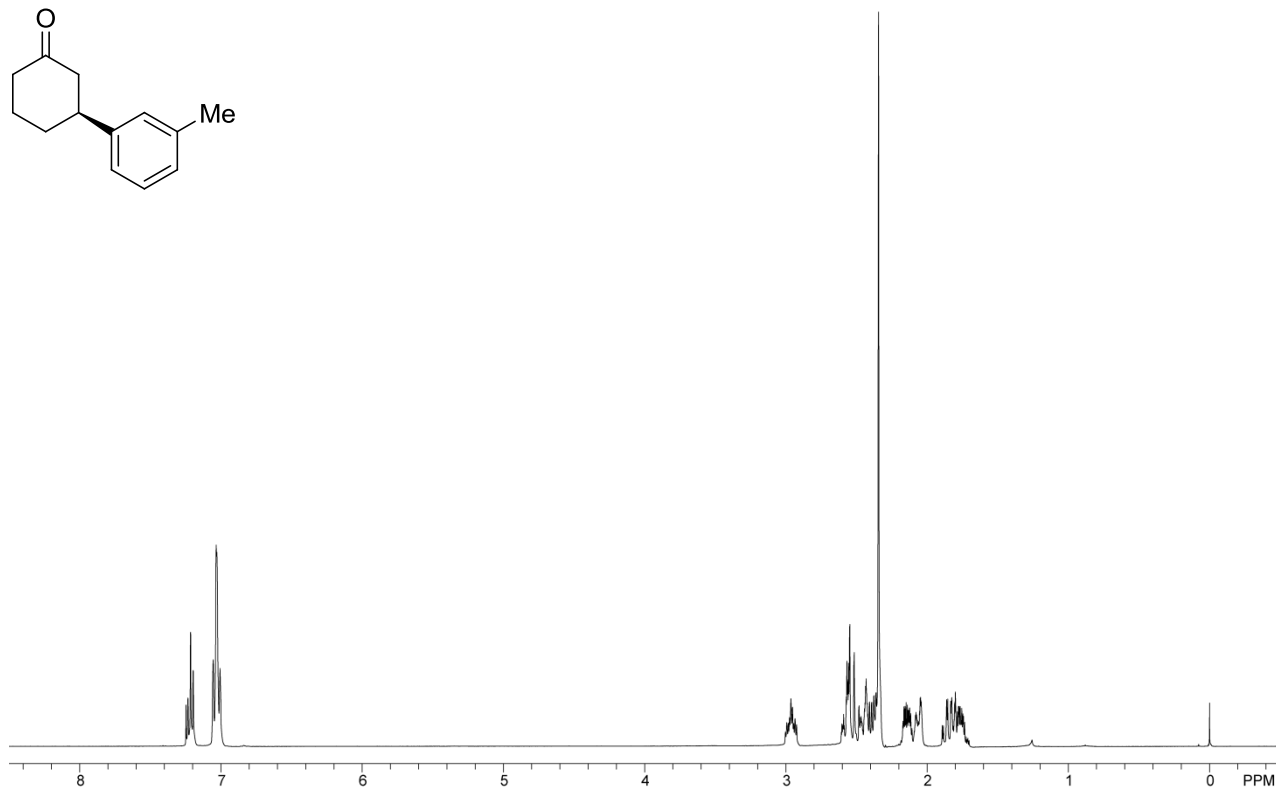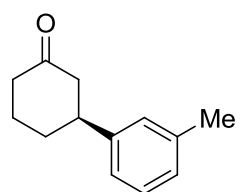
Table 2, entry 10
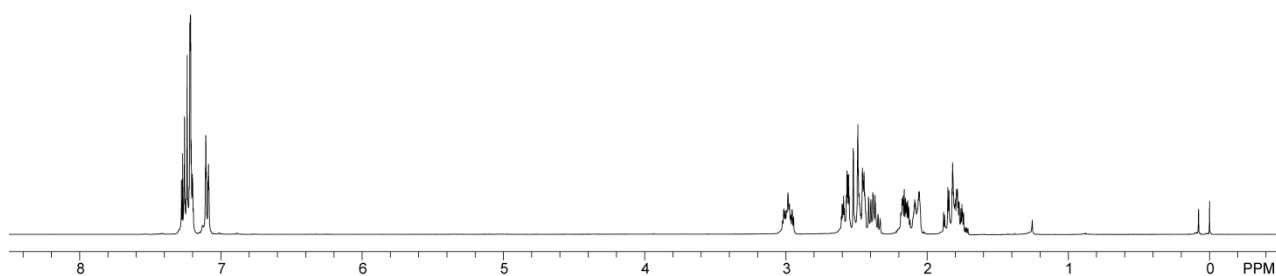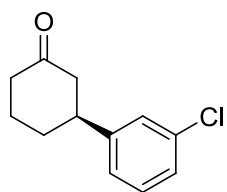
Table 2, entry 11
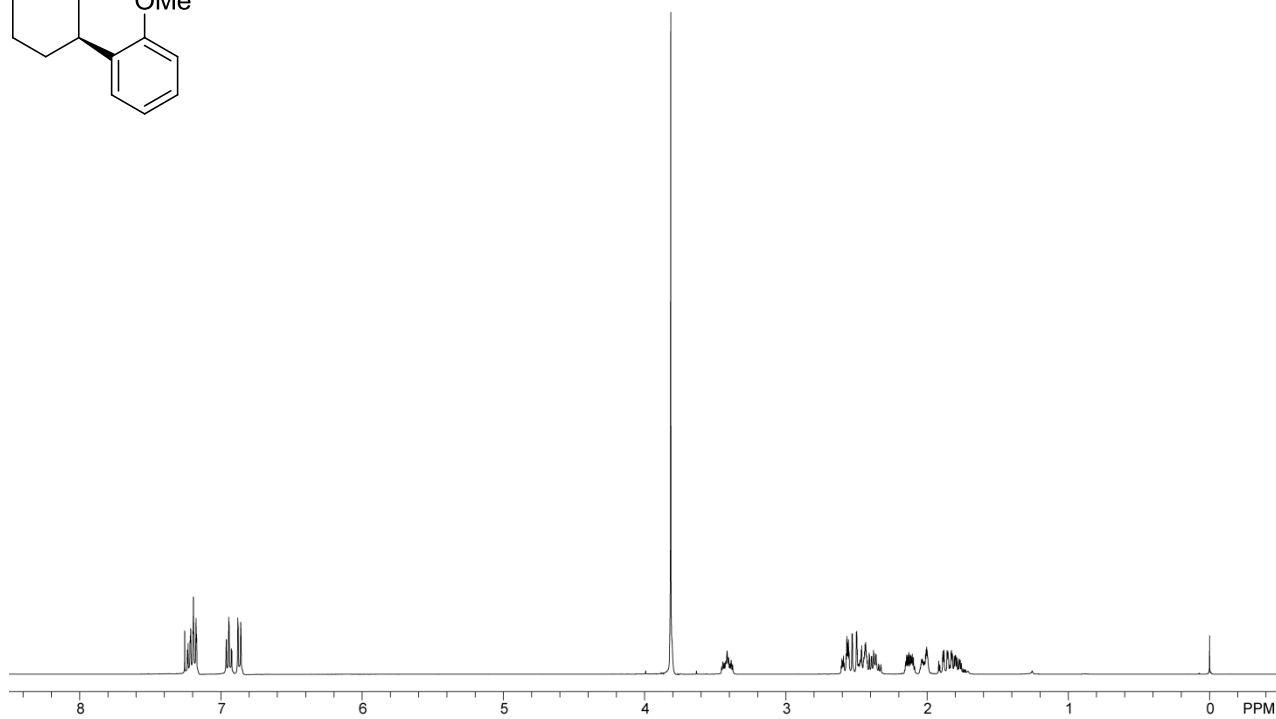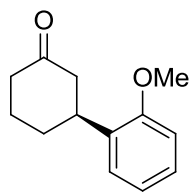
Table 2, entry 12
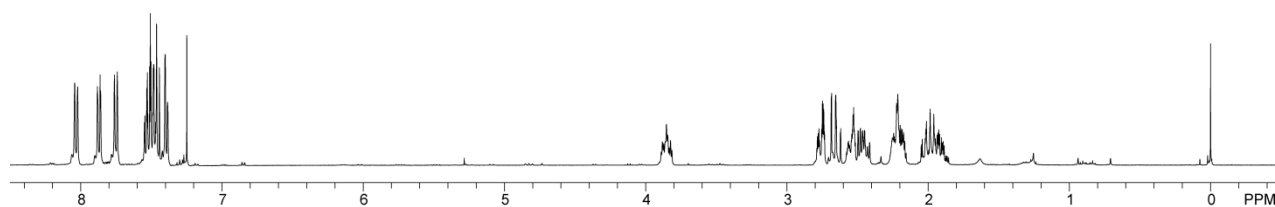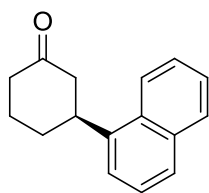
Table 2, entry 13
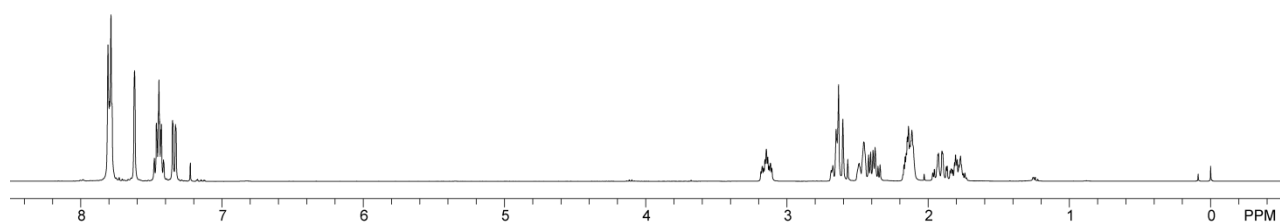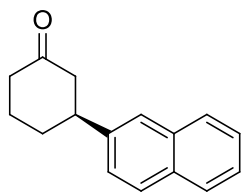
Table 2, entry 14
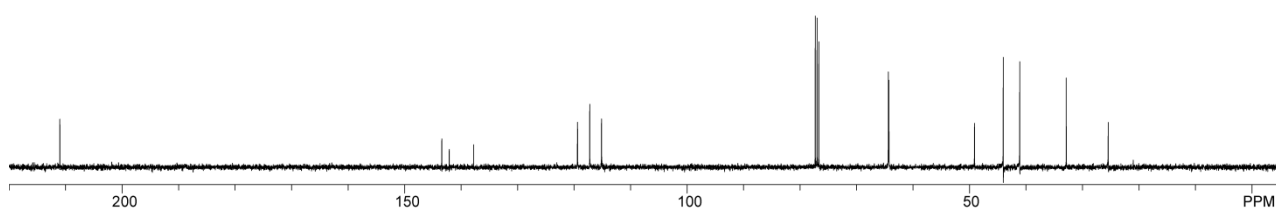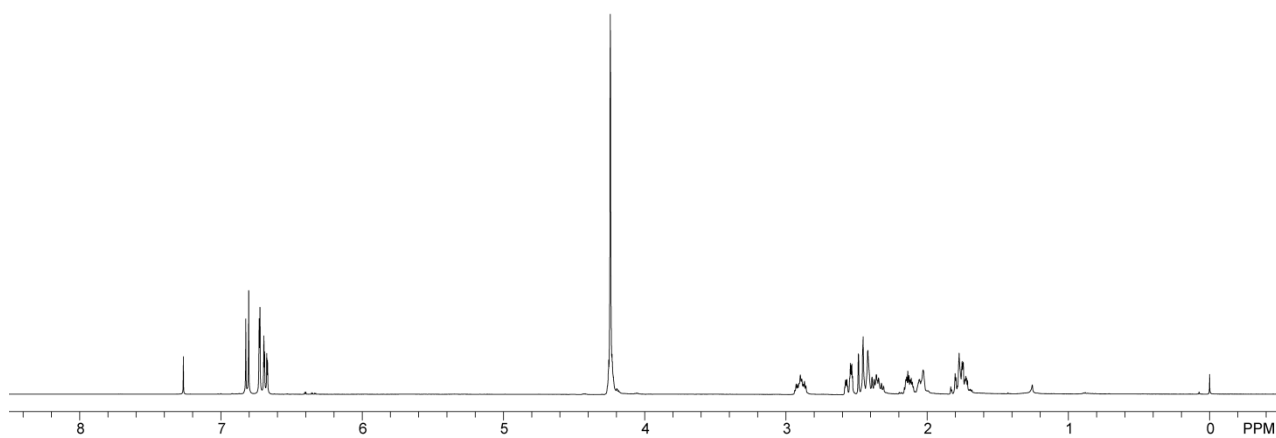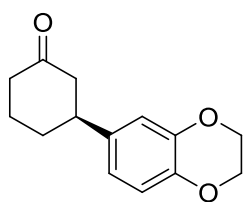
Table 2, entry 15
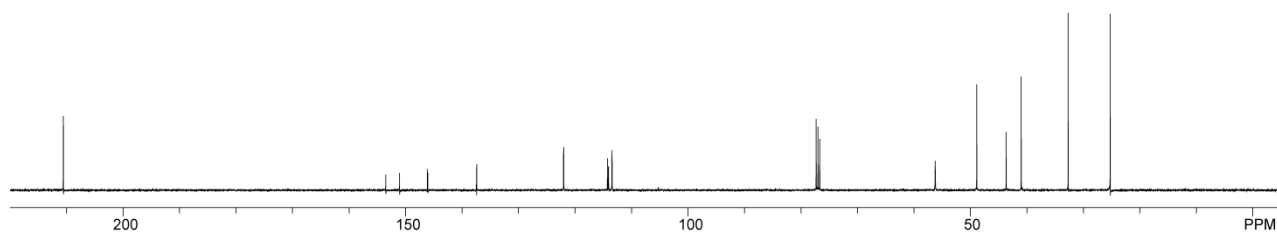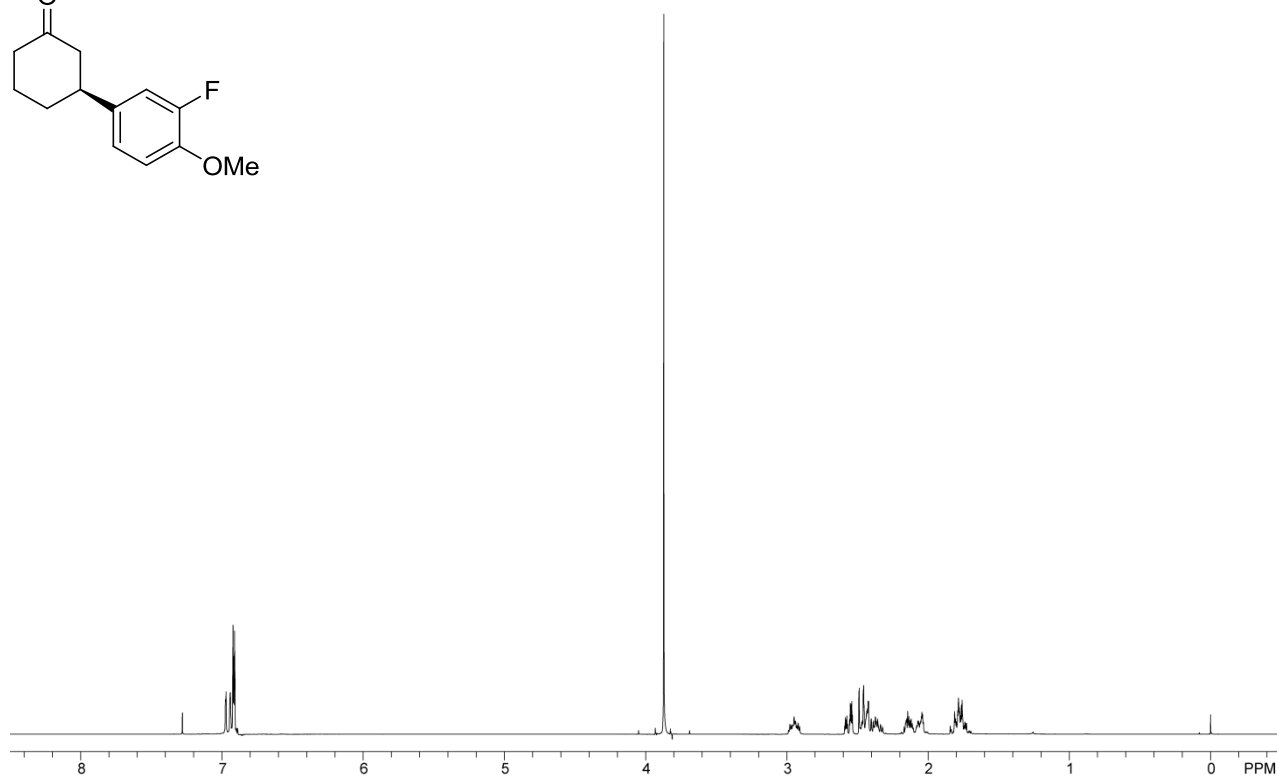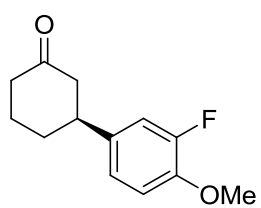
Table 2, entry 16
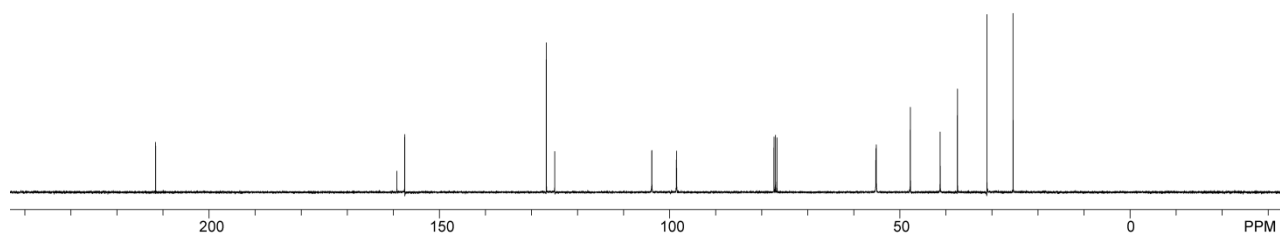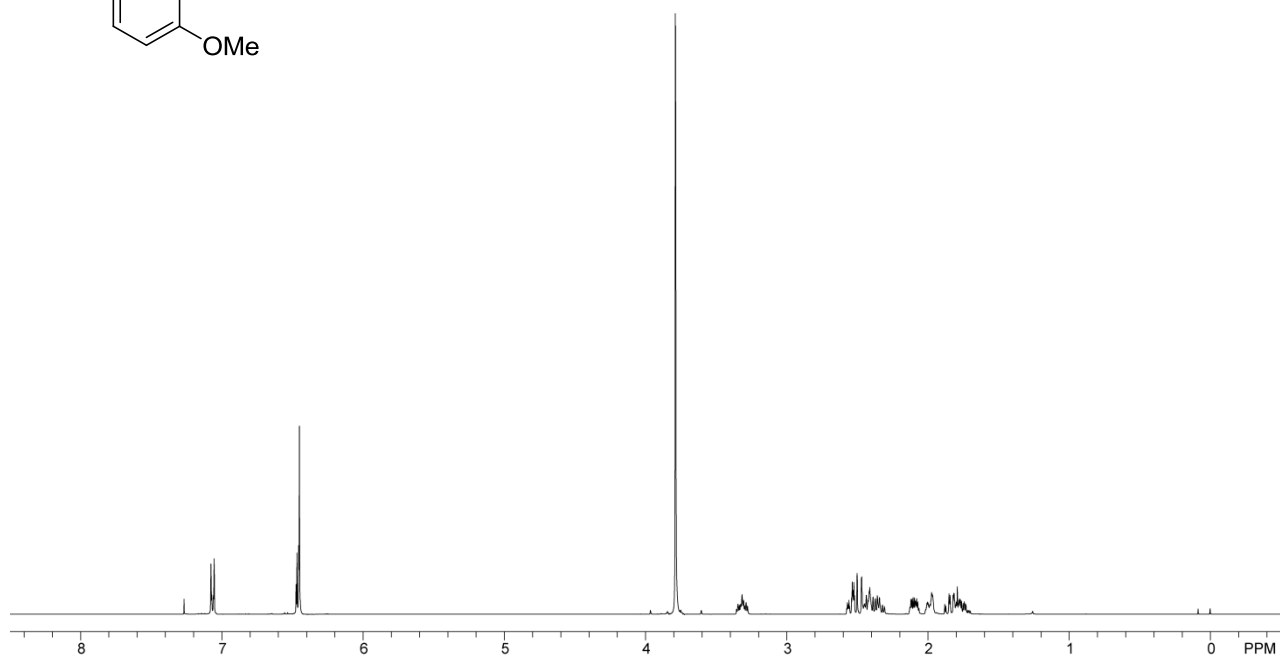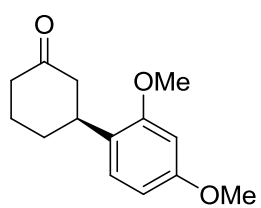
Table 2, entry 17
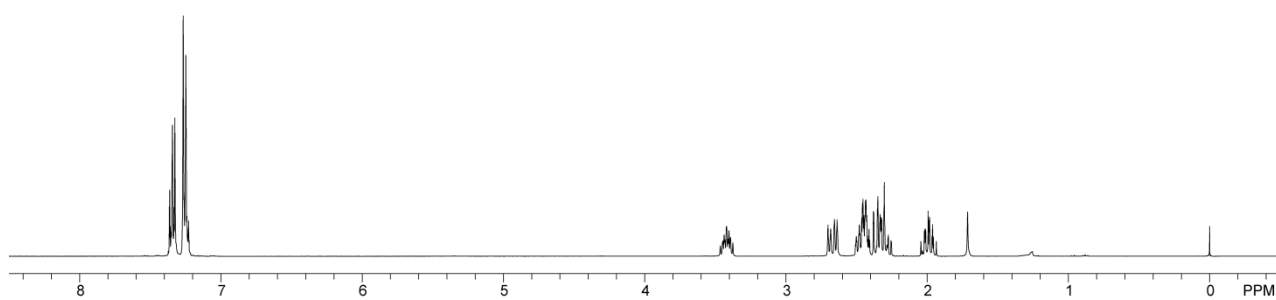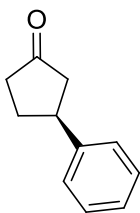
Table 2, entry 18
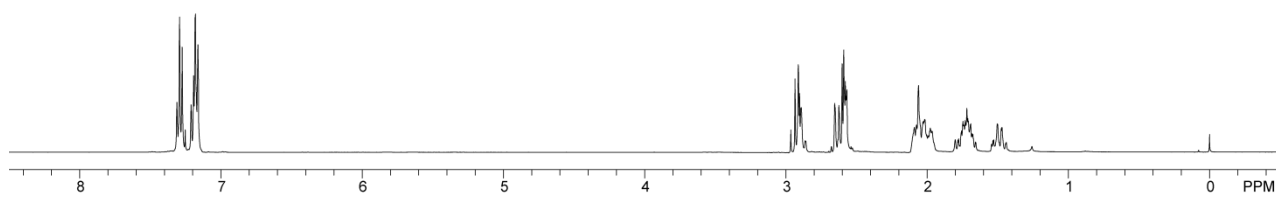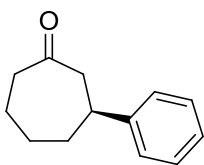
Table 2, entry 19
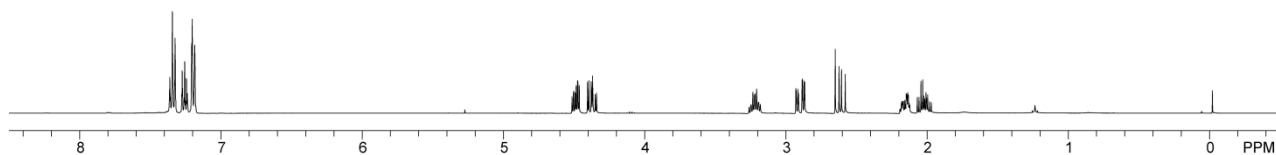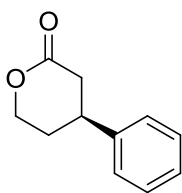
4.HPLC Diagrams for Enantiomeric Purity Determination

Table 2, entry 1

Racemic:
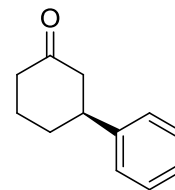
```
Acq. Operator    : LQ
Acq. Instrument : Instrument 1                    Location :    -
Injection Date  : 2010-9-14 12:38:12
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-9-14 11:39:30  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : AD-H, 1.0 ml/min, Hexane:iPrOH = 97:3
```
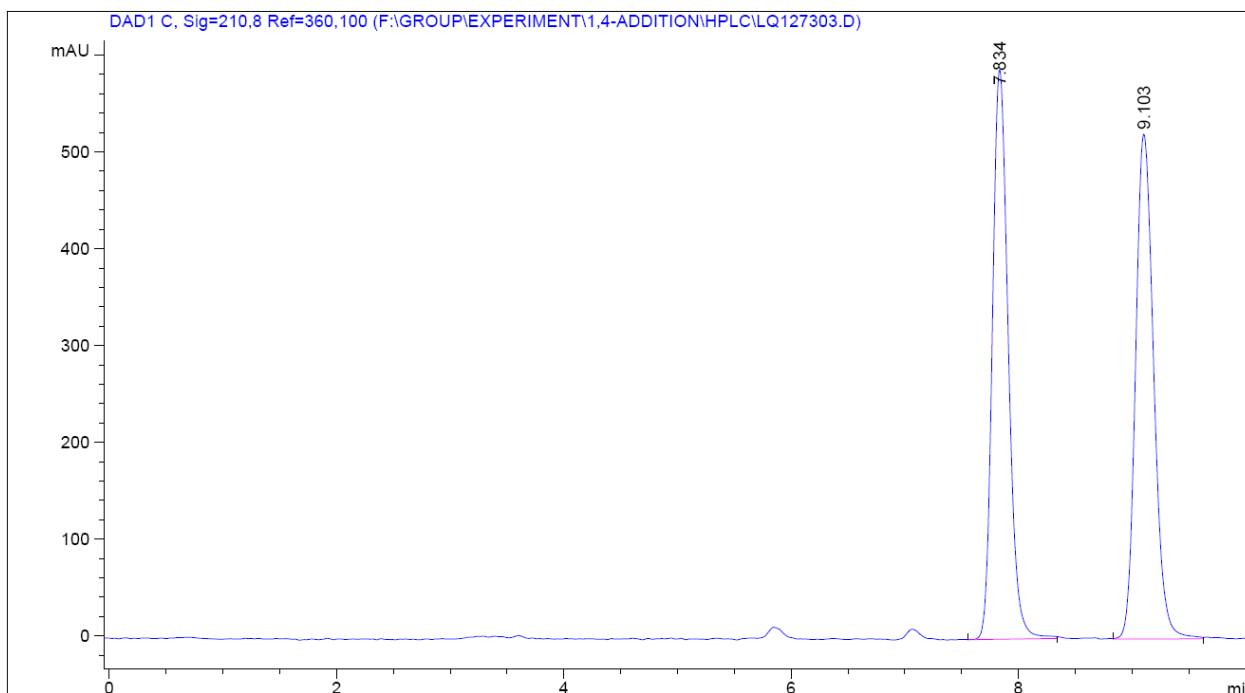


```
========================================================================
                        Area Percent Report
========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
  #   [min]        [min]   [mAU*s]      [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.834 VB    0.1507 5690.94531  589.00067  49.6197
   2   9.103 VV    0.1732 5778.17871  521.68347  50.3803

Totals :                  1.14691e4  1110.68414
```

Chiral:

Table 2, entry 1

DAD1 C, Sig=210,8 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\LQ132101.D)
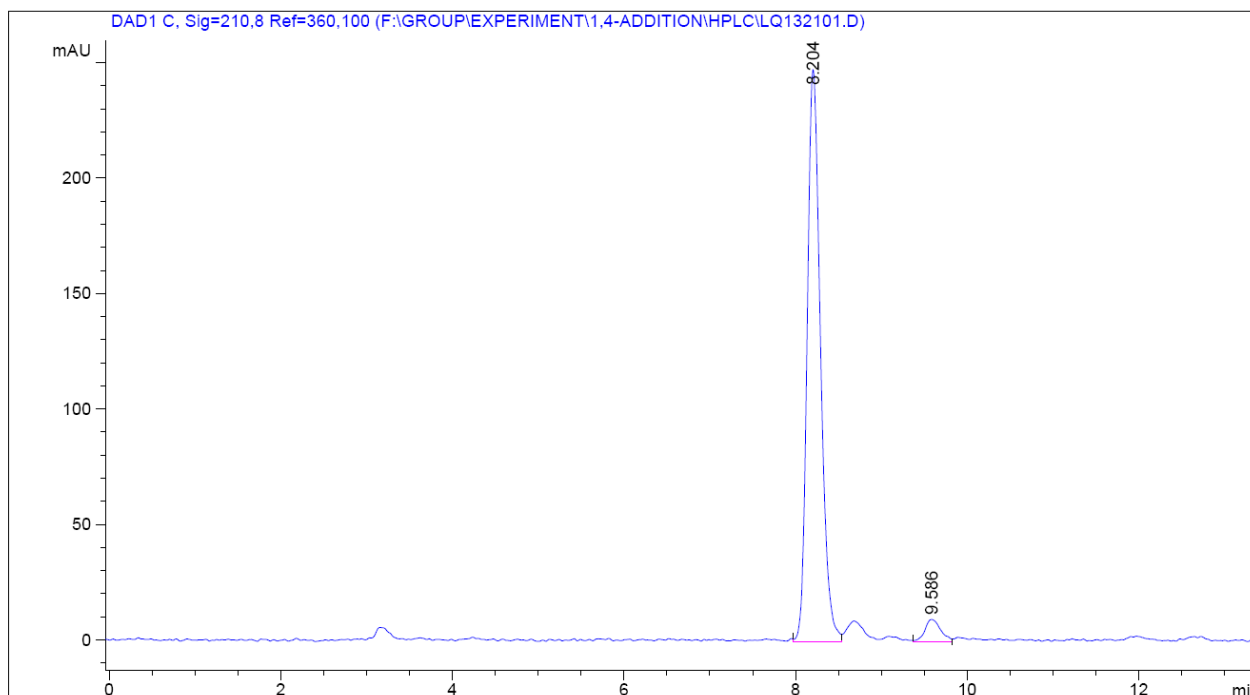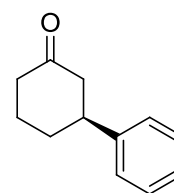
```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By            :       Signal
Multiplier           :       1.0000
Dilution             :       1.0000
Sample Amount        :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area        Height     Area
 #   [min]        [min]    [mAU*s]       [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.204 VV   0.1651 2658.40088  247.86446  95.6062
   2   9.586 BV   0.1770  122.17175    9.56810   4.3938

Totals :                  2780.57262  257.43255
```

Racemic:

```
Acq. Operator   : LQ
Acq. Instrument : Instrument 1                  Location :   -
Injection Date  : 2010-9-21 0:24:56
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-9-21 0:06:34  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : AD-H, 0.5 ml/min, Hexane:iPrOH = 97:3
```



```
======================================================================
                      Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area        Height      Area
  #   [min]        [min]   [mAU*s]       [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1  13.920 VV    0.2776 8475.13867  467.25375  48.8129
   2  14.586 VB    0.2896 8887.35938  459.57156  51.1871

Totals :                  1.73625e4   926.82532
```

Chiral:

```
Acq. Operator   : LQ
Acq. Instrument : Instrument 1              Location :   -
Injection Date  : 2010-9-21 0:41:41
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-9-21 0:06:34  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : AD-H, 0.5 ml/min, Hexane:iPrOH = 97:3
```


DAD1 C, Sig=210,8 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\LQ130100.D)

```
========================================================================
                        Area Percent Report
========================================================================

Sorted By            :      Signal
Multiplier           :      1.0000
Dilution             :      1.0000
Sample Amount        :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
  #   [min]        [min]   [mAU*s]      [mAU]       %
----|-------|----|-------|----------|----------|--------|
  1  13.832 VV   0.2639 5892.43311  337.21301  94.2444
  2  14.474 VV   0.2405  359.85989   18.53878   5.7556

Totals :                6252.29300  355.75180
```

Racemic:

Table 2, entry 3
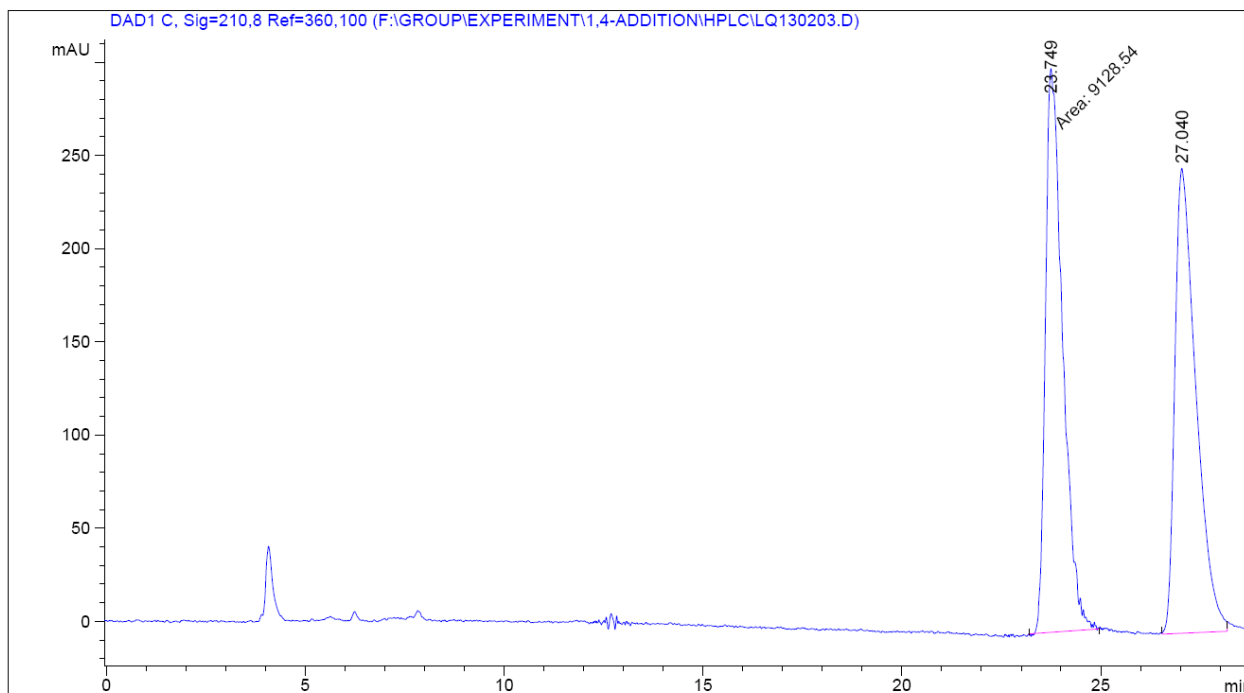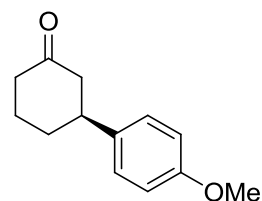

```
========================================================================
                          Area Percent Report
========================================================================

Sorted By            :        Signal
Multiplier           :        1.0000
Dilution             :        1.0000
Sample Amount        :        1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
  #   [min]        [min]   [mAU*s]      [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  23.749 MM   0.5030 9128.53613   302.46219  49.8676
   2  27.040 VV   0.4992 9177.01270   249.43385  50.1324

Totals :                  1.83055e4    551.89604
```

Chiral:

Table 2, entry 3

DAD1 C, Sig=210,8 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\LQ130301.D)
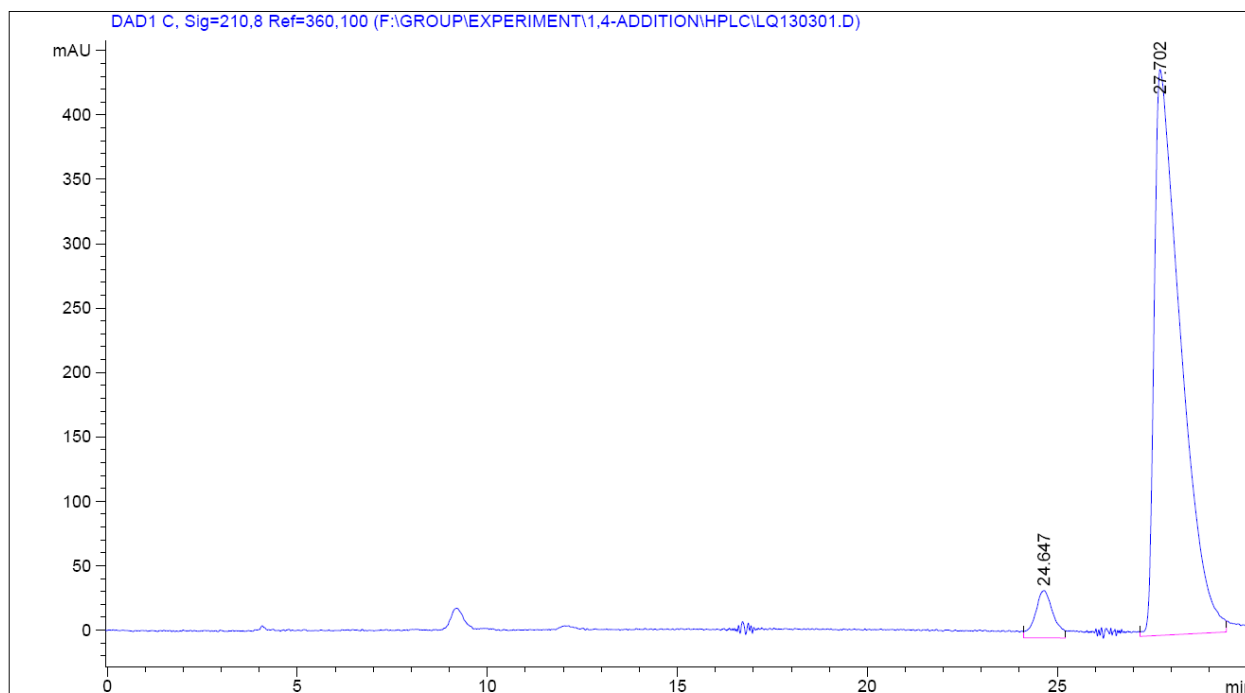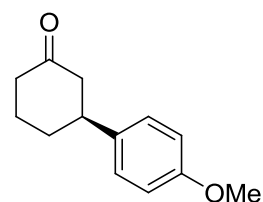

```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :       Signal
Multiplier            :       1.0000
Dilution              :       1.0000
Sample Amount         :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
  #   [min]        [min]   [mAU*s]      [mAU]        %
----|-------|----|-------|----------|----------|--------|
  1  24.647 VV    0.4273 1207.36218   36.74654   5.3748
  2  27.702 VV    0.6043 2.12562e4   439.48337  94.6252

Totals :                 2.24635e4   476.22991
```

Racemic: **Table 2, entry 4**

```
========================================================================
                         Area Percent Report
========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
  #   [min]        [min]   [mAU*s]      [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.851 VV    0.1648 1764.40417  162.32281  50.2087
   2  11.776 VV    0.2129 1749.73474  126.85610  49.7913

Totals :                  3514.13892  289.17892
```

Chiral:

```
Acq. Operator   : LQ
Acq. Instrument : Instrument 1                  Location :   -
Injection Date  : 2010-10-12 10:54:06
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-10-12 9:50:00  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : AD-H, 1.0 ml/min, Hexane:iPrOH = 97:3
```



```
=====================================================================
                     Area Percent Report
=====================================================================

Sorted By             :       Signal
Multiplier            :       1.0000
Dilution              :       1.0000
Sample Amount         :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
  #   [min]        [min]   [mAU*s]      [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   9.039 VV   0.1707 2533.46875  226.11079  94.9771
   2  12.207 VV   0.1904  133.98250    9.06477   5.0229

Totals :                 2667.45125  235.17557
```

Racemic:

Table 2, entry 5

```
=======================================================================
                        Area Percent Report
=======================================================================

Sorted By           :       Signal
Multiplier          :       1.0000
Dilution            :       1.0000
Sample Amount       :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area        Height     Area
  #   [min]        [min]   [mAU*s]       [mAU]        %
----|-------|----|-------|----------|----------|--------|
  1  18.487 MM   0.4400 2471.16846   93.59775  49.4244
  2  19.329 MM   0.4535 2528.72583   92.93226  50.5756

Totals :                4999.89429  186.53001
```

Chiral:

Table 2, entry 5

```
======================================================================
                      Area Percent Report
======================================================================

Sorted By            :       Signal
Multiplier           :       1.0000
Dilution             :       1.0000
Sample Amount        :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
 #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
  1  18.292 VV    0.2747  161.67477   7.17975   4.3439
  2  19.074 VB    0.4107 3560.21313  127.90865  95.6561

Totals :                 3721.88791  135.08841
```
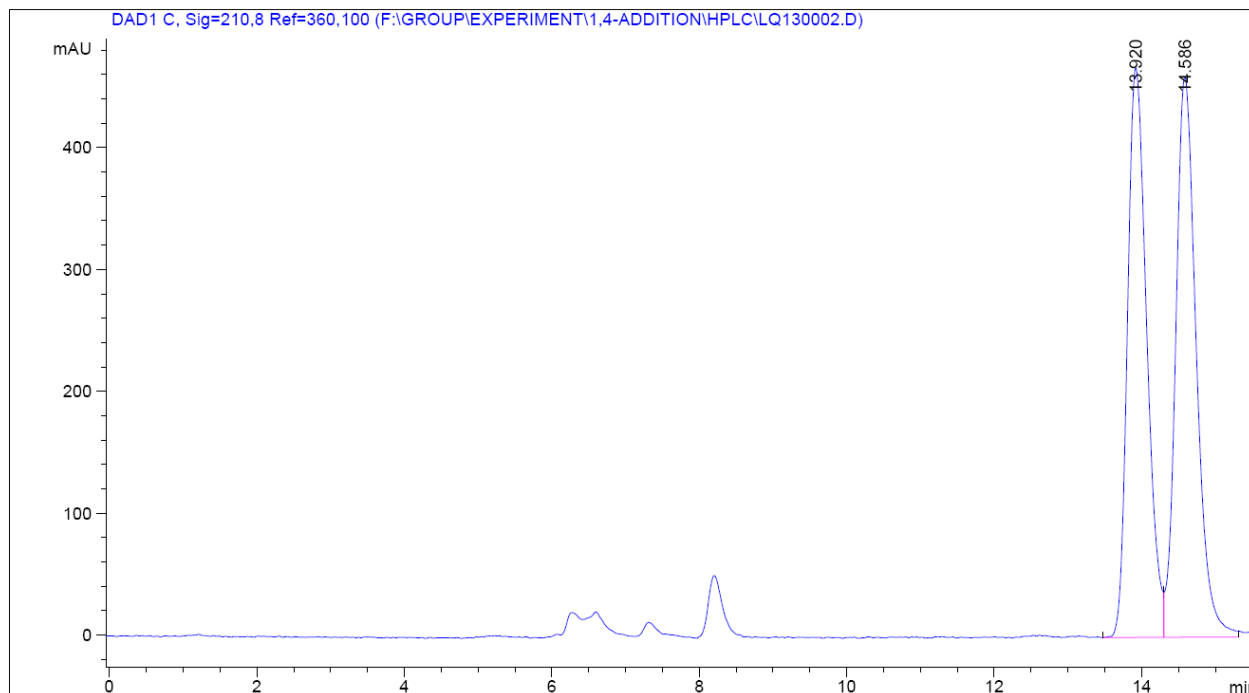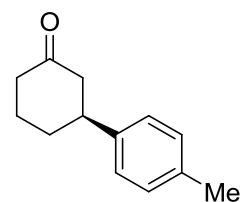
S32

Racemic:

```
Acq. Operator   : LQ
Acq. Instrument : Instrument 1                    Location :    -
Injection Date  : 2010-11-3 3:15:17
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-11-3 0:37:03   by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2010-11-3 8:28:19
                  (modified after loading)
Sample Info     : AS-H, 0.5 ml/min, Hexane:iPrOH = 60:40
```
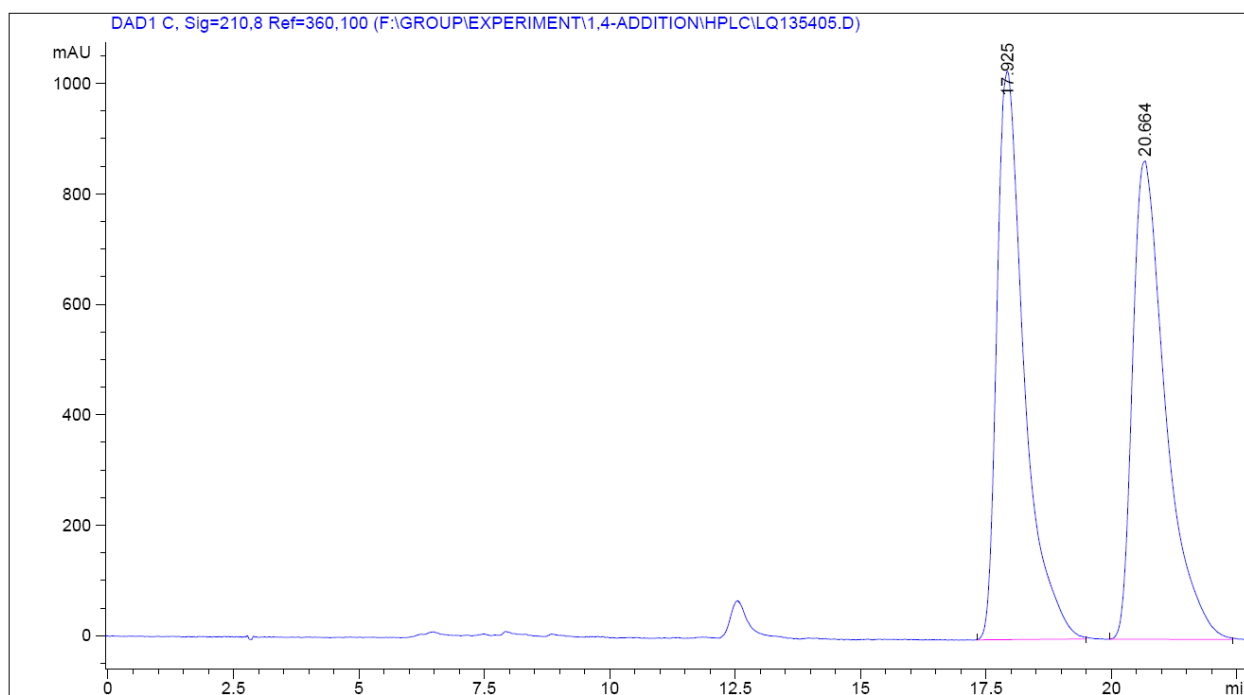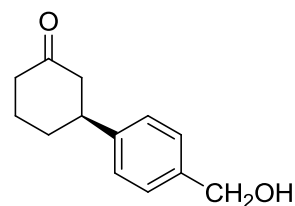


```
=====================================================================
                      Area Percent Report
=====================================================================

Sorted By            :      Signal
Multiplier           :      1.0000
Dilution             :      1.0000
Sample Amount        :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
  #   [min]        [min]   [mAU*s]      [mAU]       %
----|-------|----|-------|----------|----------|--------|
  1  17.925 BB    0.5427 3.90569e4   1029.36072  49.7646
  2  20.664 BB    0.6372 3.94265e4    865.85986  50.2354

Totals :                 7.84834e4   1895.22058
```
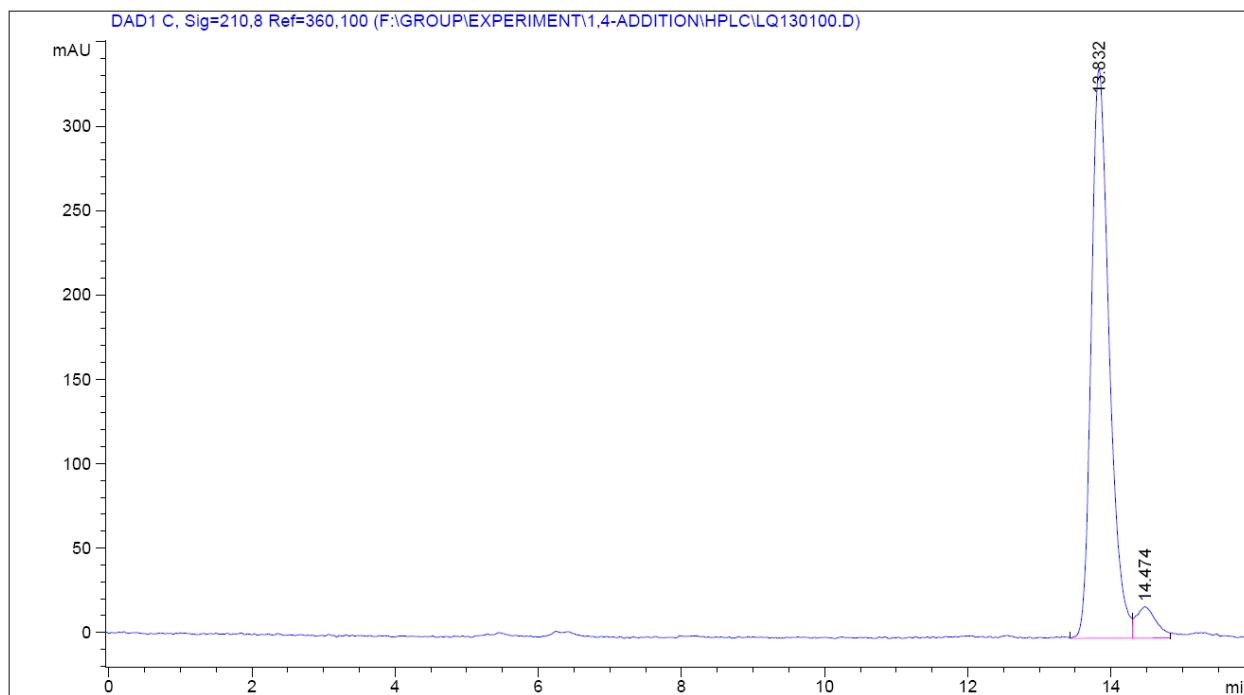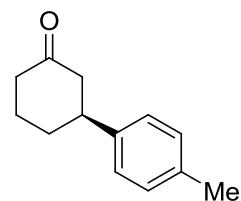
Chiral:

```
Acq. Operator    : LQ
Acq. Instrument  : Instrument 1                    Location :    -
Injection Date   : 2010-11-3 4:05:03
Acq. Method      : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed     : 2010-11-3 0:37:03  by LQ
                   (modified after loading)
Analysis Method  : C:\Chem32\1\METHODS\DEF_LC.M
Last changed     : 2010-11-3 8:28:19
                   (modified after loading)
Sample Info      : AS-H, 0.5 ml/min, Hexane:iPrOH = 60:40
```


DAD1 C, Sig=210,8 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\LQ135501.D)

```
======================================================================
                       Area Percent Report
======================================================================

Sorted By             :       Signal
Multiplier            :       1.0000
Dilution              :       1.0000
Sample Amount         :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area        Height      Area
  #   [min]        [min]    [mAU*s]      [mAU]        %
----|-------|----|-------|----------|-----------|--------|
  1  18.051  BB   0.5251 7924.88477   228.23091   95.9818
  2  20.869  BB   0.4082  331.76682    10.34498    4.0182

Totals :                  8256.65158  238.57589
```

Racemic:

```
======================================================================
                        Area Percent Report
======================================================================

Sorted By           :       Signal
Multiplier          :       1.0000
Dilution            :       1.0000
Sample Amount       :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area        Height     Area
 #   [min]        [min]    [mAU*s]       [mAU]       %
----|-------|----|-------|----------|----------|--------|
  1  24.563 BB    0.4660 7363.28027   241.28400  49.8687
  2  28.379 BB    0.5627 7402.04443   173.87190  50.1313

Totals :                  1.47653e4    415.15590
```

Chiral:

Table 2, entry 7

DAD1 C, Sig=210,8 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\LQ135700.D)

28.323
Area: 10384.5

24.750

```
=====================================================================
                      Area Percent Report
=====================================================================

Sorted By           :      Signal
Multiplier          :      1.0000
Dilution            :      1.0000
Sample Amount       :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area        Height     Area
 #   [min]        [min]    [mAU*s]       [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  24.750 BB   0.4908  746.37268   18.97841   6.7054
   2  28.323 MM   0.7495 1.03845e4   230.92516  93.2946

Totals :                 1.11308e4   249.90357
```
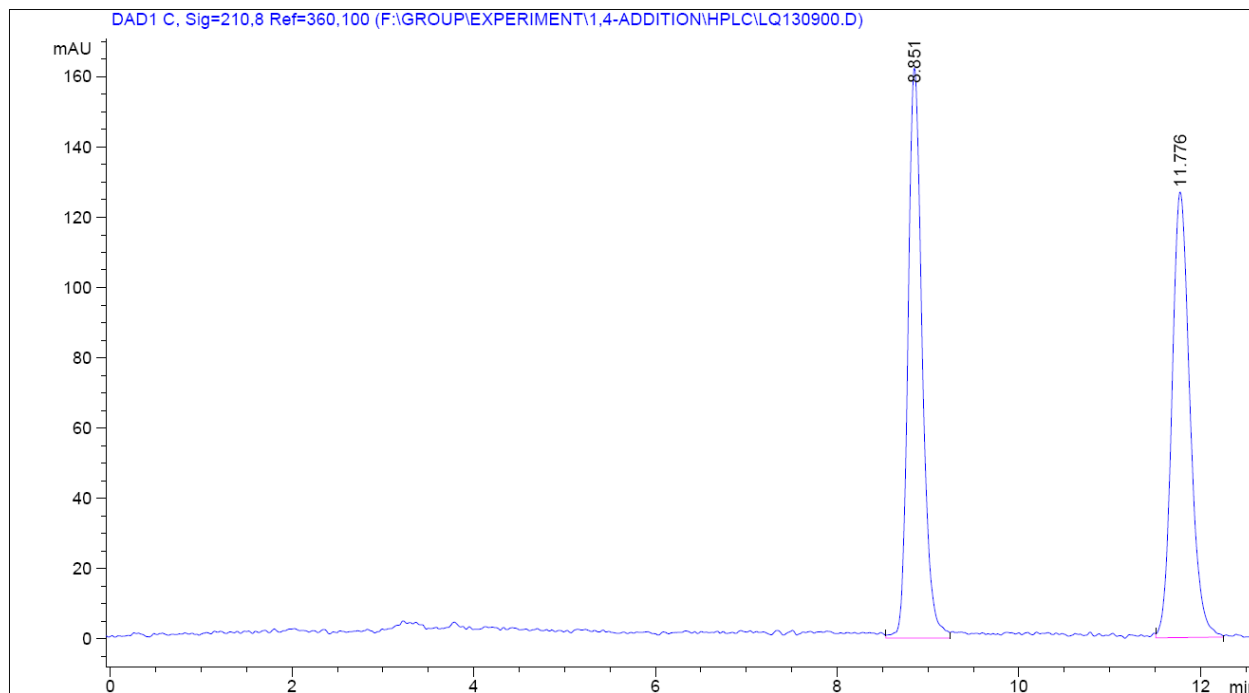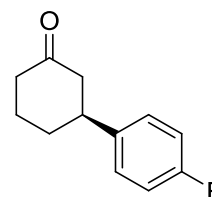
Racemic:

Table 2, entry 8

DAD1 C, Sig=210,8 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\LQ136600.D)

```
=====================================================================
                      Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
  1  30.560 VB   0.5437 8132.50195  217.77353  49.7331
  2  32.917 VV   0.5883 8219.79688  179.49841  50.2669

Totals :                  1.63523e4   397.27194
```
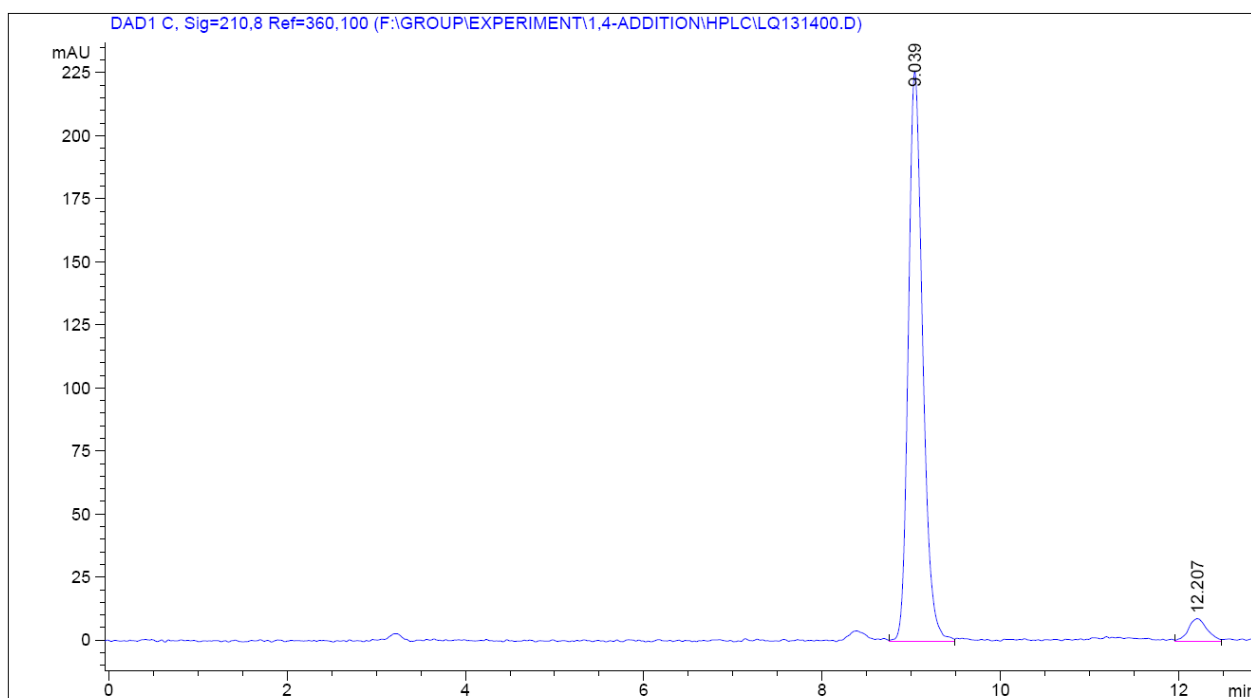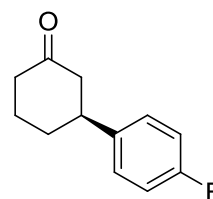
S37

Chiral:

Table 2, entry 8


```
Acq. Operator    : SZB
Acq. Instrument  : Instrument 1              Location :    -
Injection Date   : 2010-11-4 11:12:52
Acq. Method      : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed     : 2010-11-4 10:15:39  by SZB
                   (modified after loading)
Analysis Method  : C:\Chem32\1\METHODS\DEF_LC.M
Last changed     : 2004-4-7 0:10:12
Sample Info      : AD-H, 0.5 ml/min, Hexane:iPrOH = 90:10
```



```
=====================================================================
                      Area Percent Report
=====================================================================

Sorted By             :       Signal
Multiplier            :       1.0000
Dilution              :       1.0000
Sample Amount         :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height    Area
  #   [min]        [min]   [mAU*s]      [mAU]       %
----|-------|----|-------|----------|----------|--------|
  1  30.724  VV   0.4405 1978.62354   54.37933   6.7239
  2  32.676  VV   0.7074 2.74483e4   504.49567  93.2761

Totals :                  2.94269e4   558.87499
```
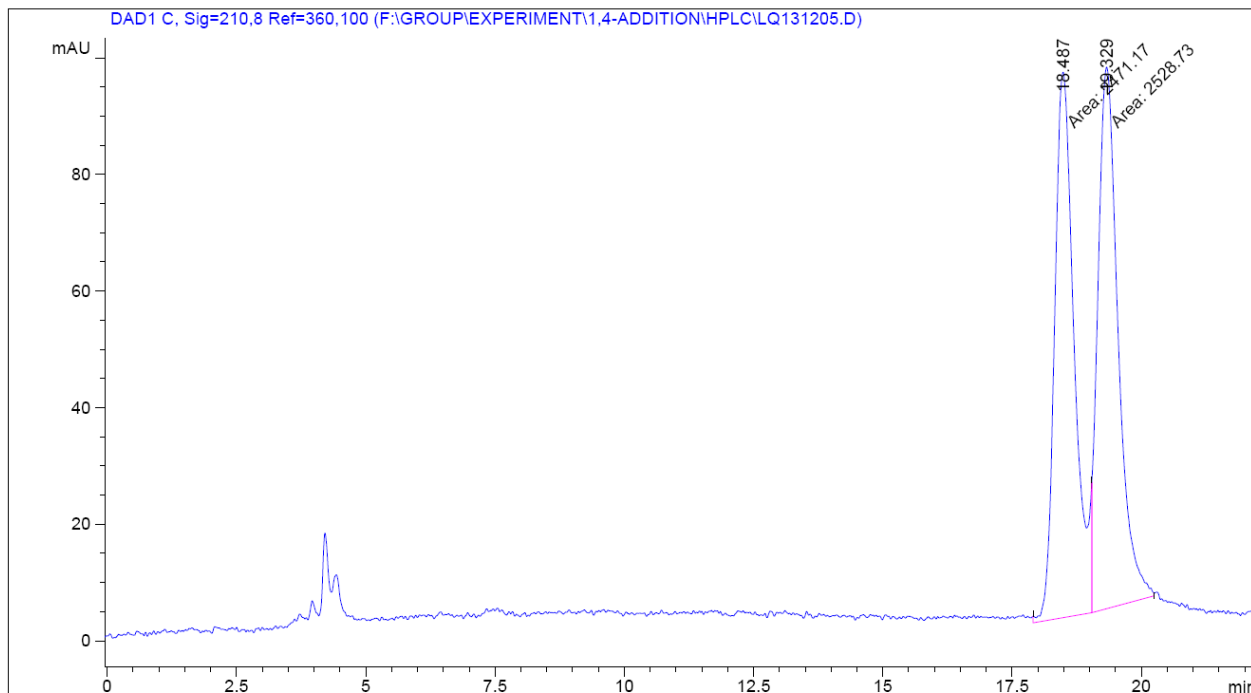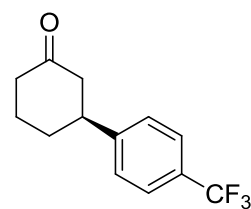
Racemic:

```
Acq. Operator    : LQ
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 2010-10-12 12:01:21
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-10-12 11:48:25  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : AD-H, 0.9 ml/min, Hexane:iPrOH = 98:2
```



```
====================================================================
                       Area Percent Report
====================================================================

Sorted By            :      Signal
Multiplier           :      1.0000
Dilution             :      1.0000
Sample Amount        :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area        Height     Area
  #   [min]        [min]   [mAU*s]       [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.247 VV   0.1620 2995.16040  281.88080  49.6263
   2   8.780 VV   0.1717 3040.27466  269.42300  50.3737

Totals :                 6035.43506  551.30380
```
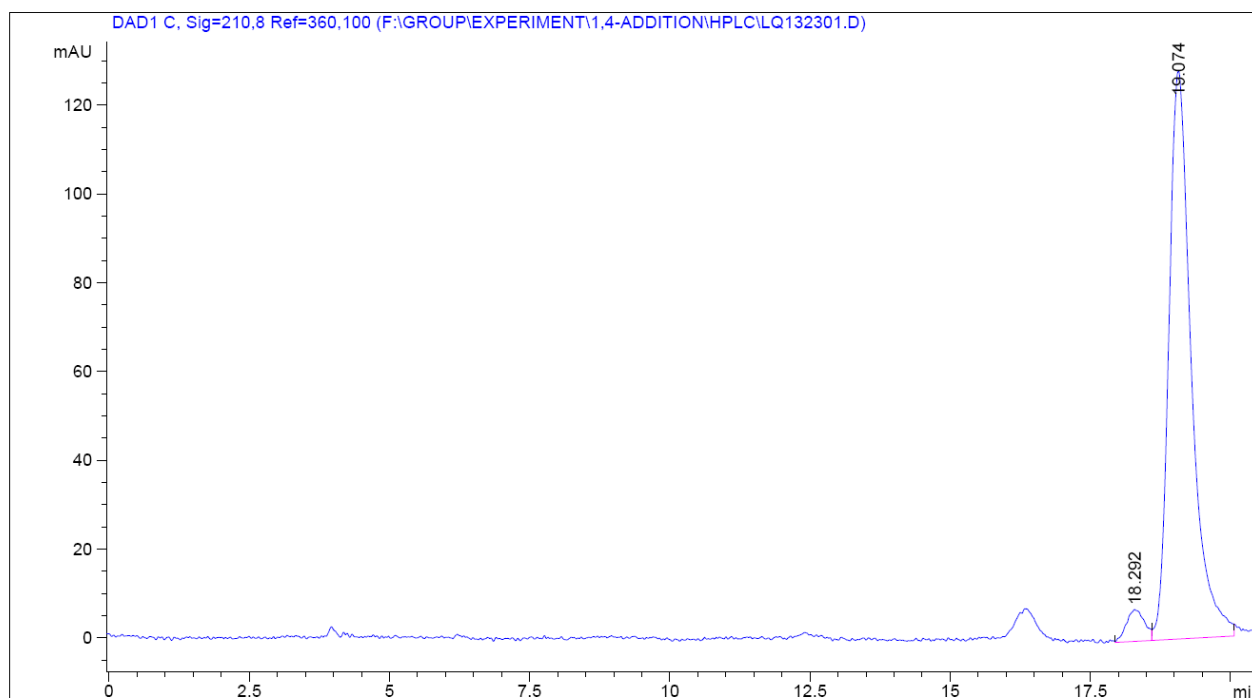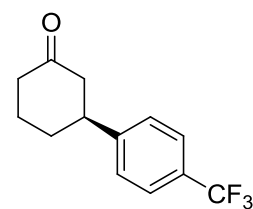
Chiral:

Table 2, entry 9


```
========================================================================
                       Area Percent Report
========================================================================

Sorted By             :       Signal
Multiplier            :       1.0000
Dilution              :       1.0000
Sample Amount         :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
  #   [min]        [min]   [mAU*s]      [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1   8.147 VV   0.1655  2735.45264  250.33942  94.7847
   2   8.668 VV   0.1692   150.51089   12.09321   5.2153

Totals :                  2885.96353  262.43263
```

Racemic:

```
Acq. Operator   : LQ
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 2010-10-19 3:17:19
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-10-19 2:53:56  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : AD-H, 0.9 ml/min, Hexane:iPrOH = 93:7
```



```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :       Signal
Multiplier            :       1.0000
Dilution              :       1.0000
Sample Amount         :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area        Height     Area
  #    [min]       [min]   [mAU*s]       [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.335 VV    0.1521 2046.60547  209.17383  50.0521
   2   8.085 BBA   0.1611 2042.34216  193.58163  49.9479

Totals :                  4088.94763  402.75546
```
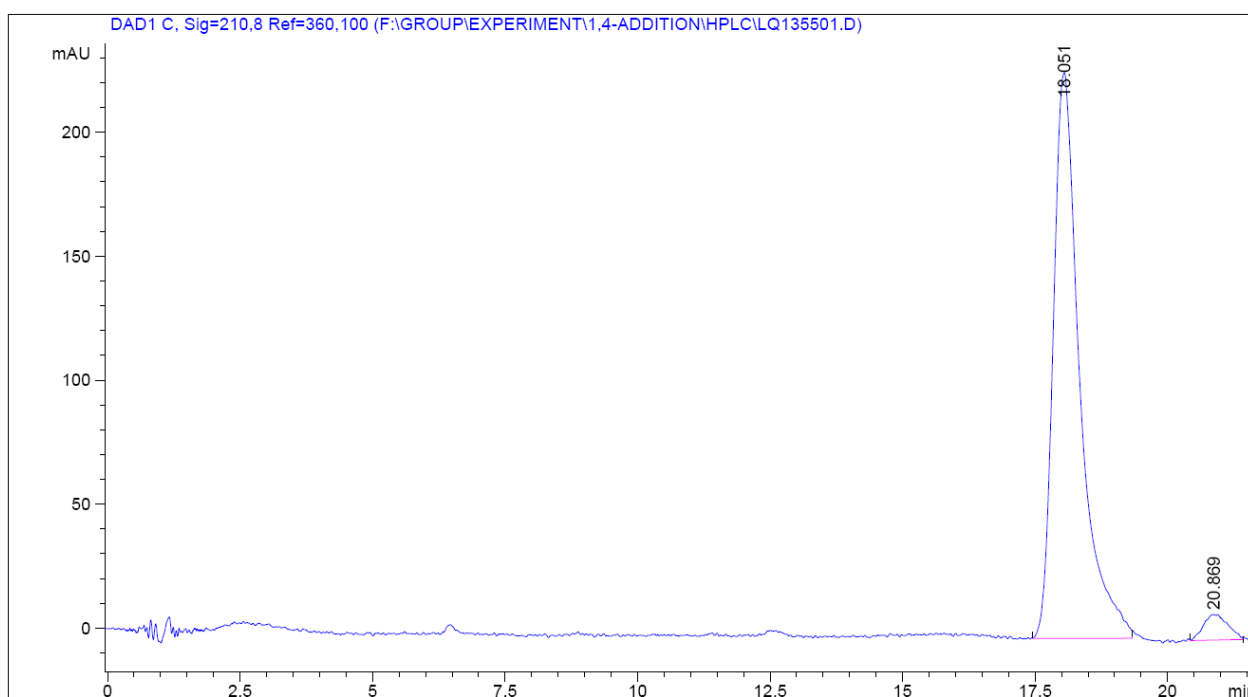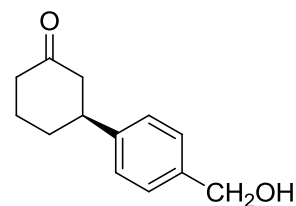
Chiral:

```
Acq. Operator   : LQ
Acq. Instrument : Instrument 1              Location :   -
Injection Date  : 2010-10-19 3:06:17
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-10-19 2:53:56  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : AD-H, 0.9 ml/min, Hexane:iPrOH = 93:7
```



```
=====================================================================
                      Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
  #   [min]        [min]    [mAU*s]      [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.328 MM   0.1682 3131.67456  310.29959  93.8196
   2   8.077 VV   0.1650  206.30066   17.57262   6.1804

Totals :                 3337.97522  327.87222
```
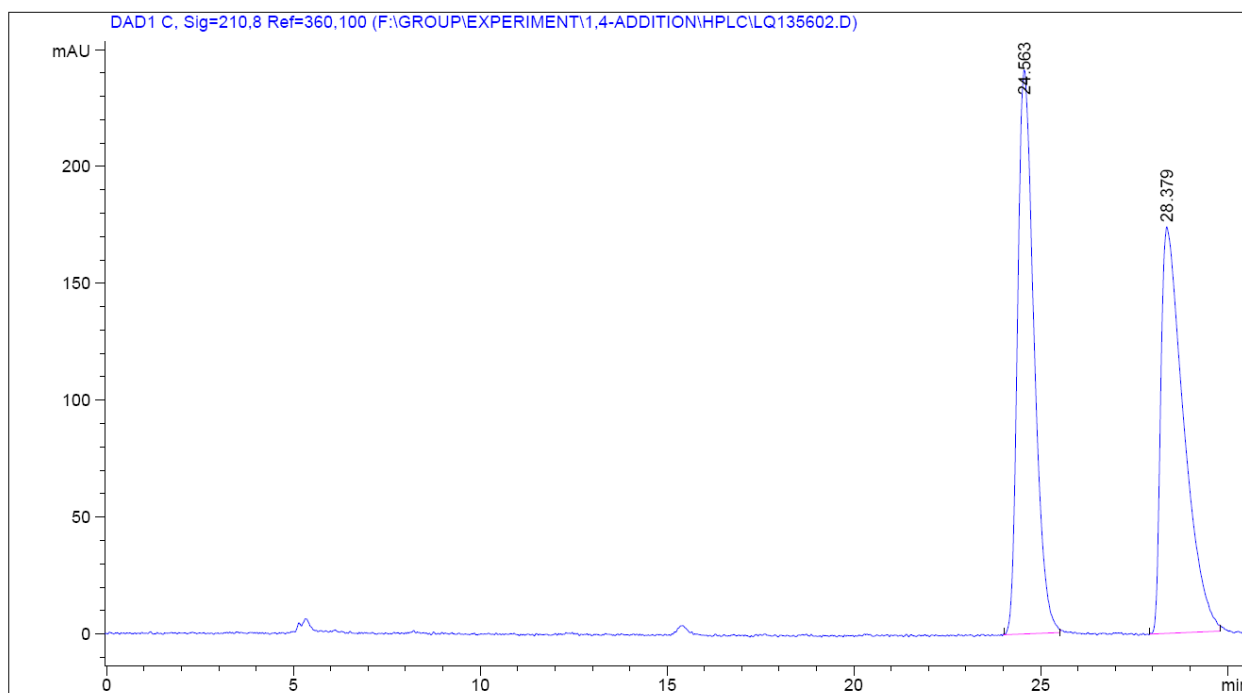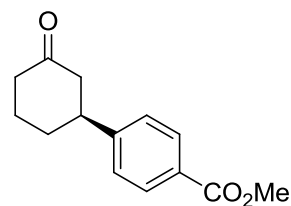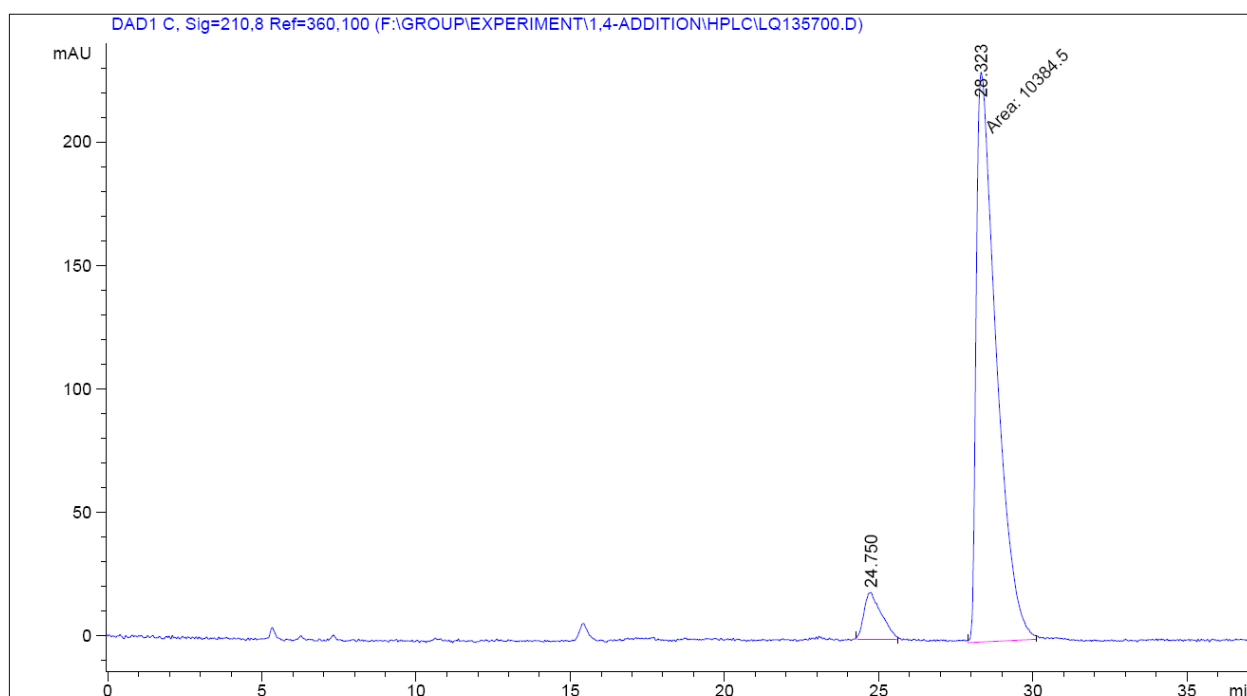
Racemic:

Table 2, entry 11

```
=====================================================================
                       Area Percent Report
=====================================================================

Sorted By            :      Signal
Multiplier           :      1.0000
Dilution             :      1.0000
Sample Amount        :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type   Width     Area        Height     Area
  #   [min]        [min]   [mAU*s]       [mAU]        %
----|-------|----|-------|----------|----------|--------|
  1  12.998 VB    0.2422 5463.16113  345.96738  49.9448
  2  14.214 BB    0.2632 5475.24414  320.70224  50.0552

Totals :                  1.09384e4   666.66962
```
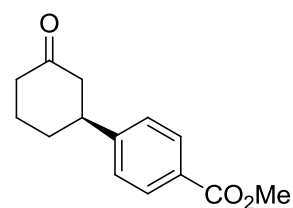
Chiral:

```
Acq. Operator    : LQ
Acq. Instrument : Instrument 1                  Location :   -
Injection Date  : 2010-10-19 11:14:55
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-10-19 9:20:33  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : AD-H, 0.7 ml/min, Hexane:iPrOH = 97:3
```


DAD1 C, Sig=210,8 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\LQ133502.D)

```
======================================================================
                    Area Percent Report
======================================================================

Sorted By             :       Signal
Multiplier            :       1.0000
Dilution              :       1.0000
Sample Amount         :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area        Height     Area
  #   [min]        [min]   [mAU*s]       [mAU]       %
----|-------|----|-------|----------|-----------|--------|
  1  13.085  VV   0.2433  2544.67310  162.00406  94.3367
  2  14.293  VV   0.2105   152.76283    9.06987   5.6633

Totals :                  2697.43593  171.07393
```
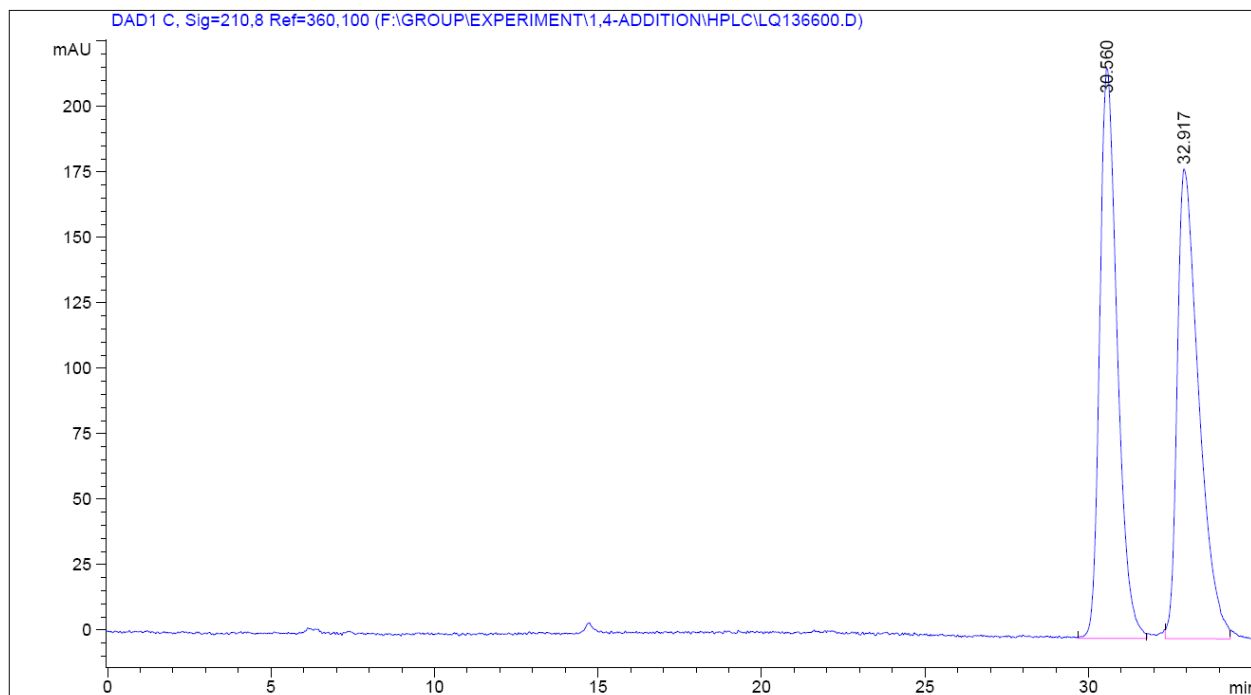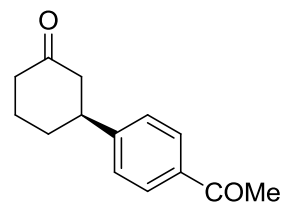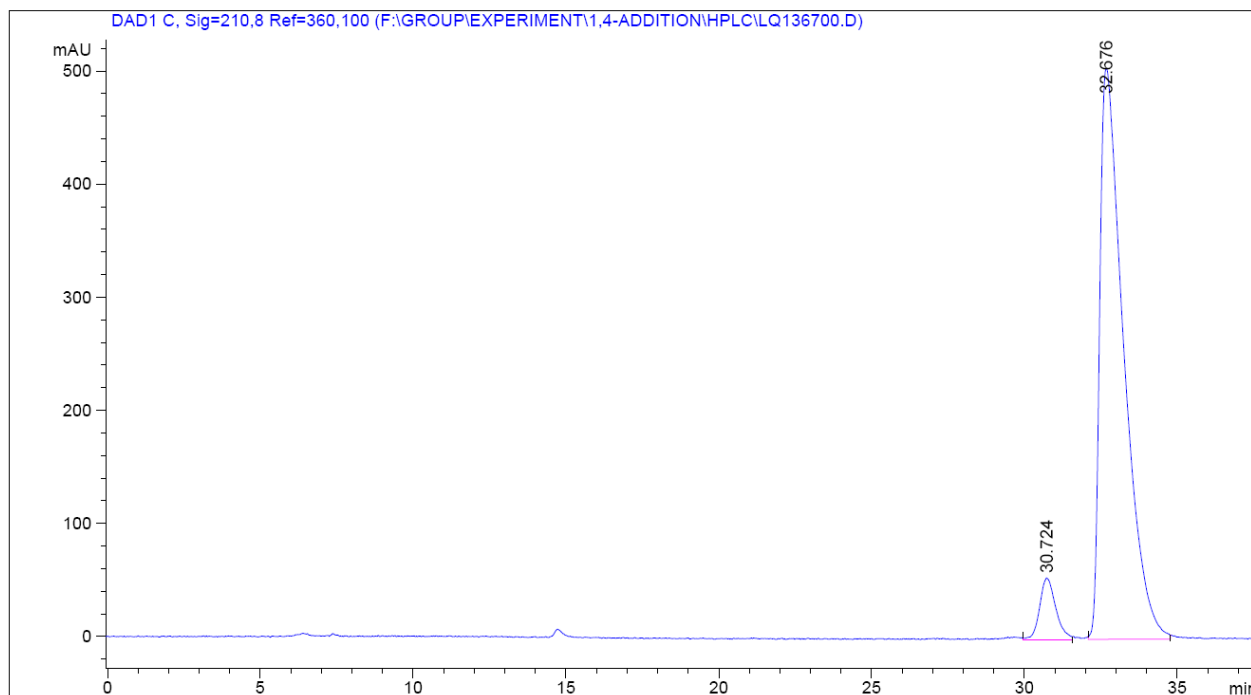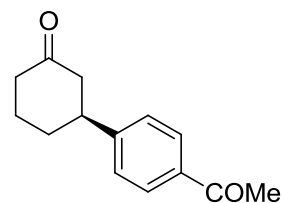
Racemic:

Table 2, entry 12

Acq. Operator   : LQ
Acq. Instrument : Instrument 1   Location :   -
Injection Date   : 2010-10-16 8:04:37
Acq. Method   : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed   : 2010-10-16 4:18:24   by LQ
                   (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed   : 2004-4-7 0:10:12
Sample Info   : OJ-H, 0.5 ml/min, Hexane:iPrOH = 95:5
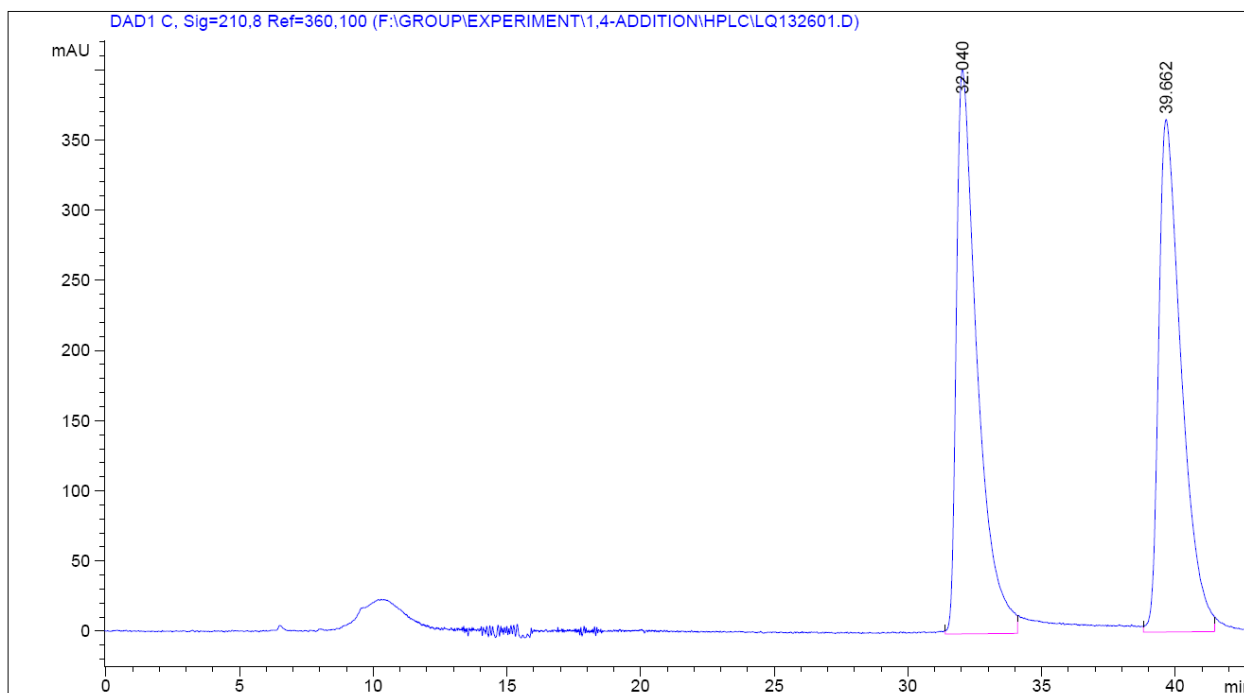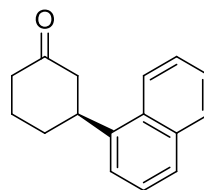
```
=====================================================================
                     Area Percent Report
=====================================================================

Sorted By            :      Signal
Multiplier           :      1.0000
Dilution             :      1.0000
Sample Amount        :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area        Height      Area
  #   [min]        [min]    [mAU*s]      [mAU]        %
----|-------|----|-------|----------|----------|--------|
  1  32.040 VV   0.7141 2.17902e4   402.16553  50.2235
  2  39.662 VV   0.7979 2.15962e4   365.22809  49.7765

Totals :                 4.33864e4   767.39362
```
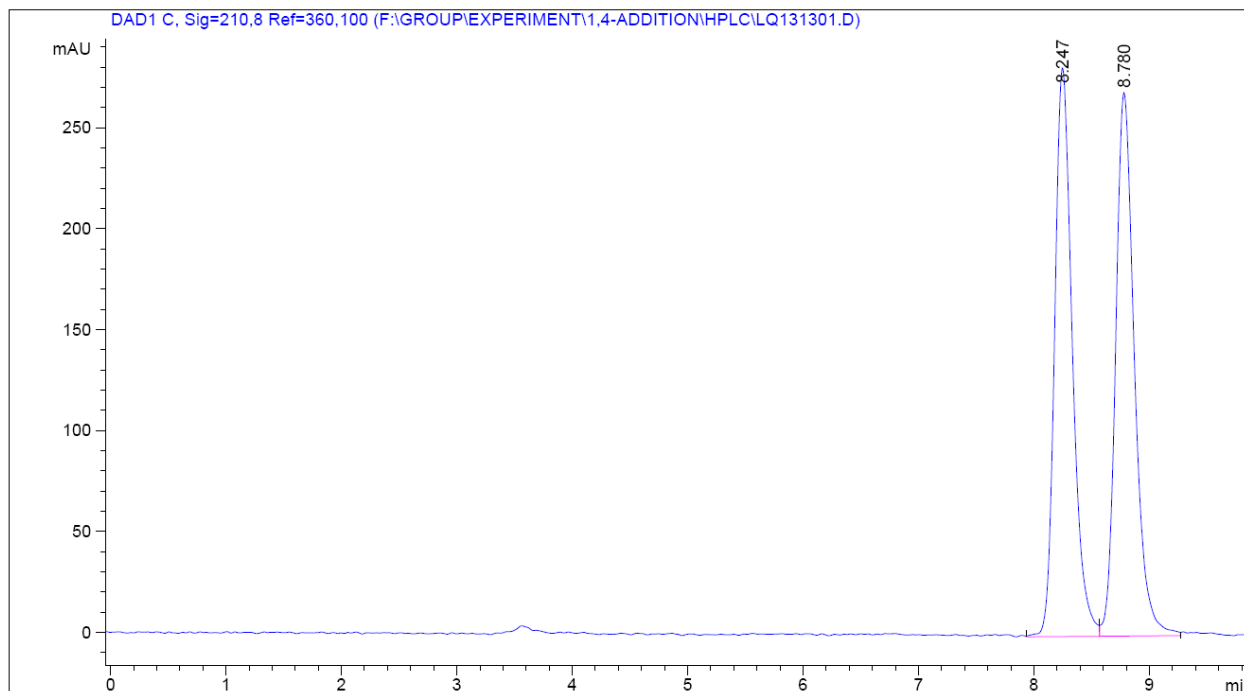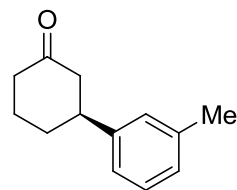
Chiral:

Table 2, entry 12

Acq. Operator   : LQ
Acq. Instrument : Instrument 1   Location :   -
Injection Date : 2010-10-16 8:49:09
Acq. Method   : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed   : 2010-10-16 4:18:24 by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed   : 2004-4-7 0:10:12
Sample Info   : OJ-H, 0.5 ml/min, Hexane:iPrOH = 95:5

DAD1 C, Sig=210,8 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\LQ132702.D)
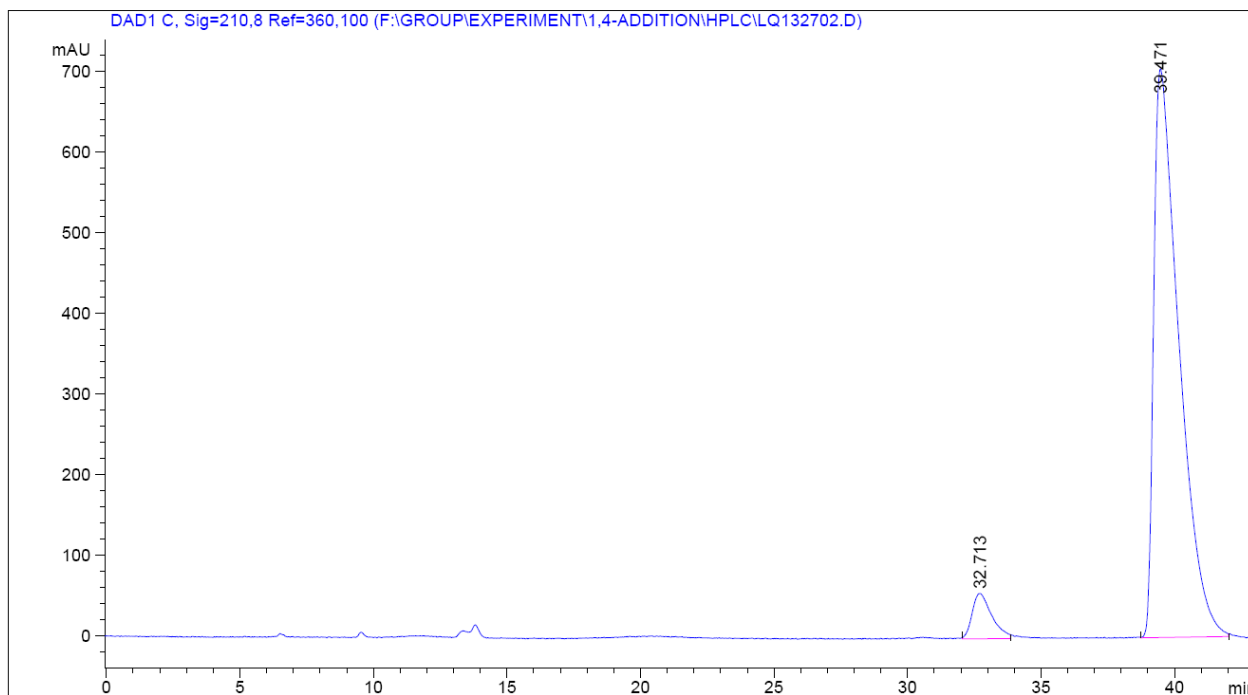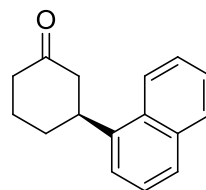

```
====================================================================
                       Area Percent Report
====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
 #   [min]        [min]   [mAU*s]      [mAU]       %
---|-------|----|-------|----------|----------|--------|
  1  32.713 VV    0.6006 2855.08960   56.20997   5.8047
  2  39.471 BV    0.7725 4.63311e4   705.19165  94.1953

Totals :                  4.91861e4   761.40162
```
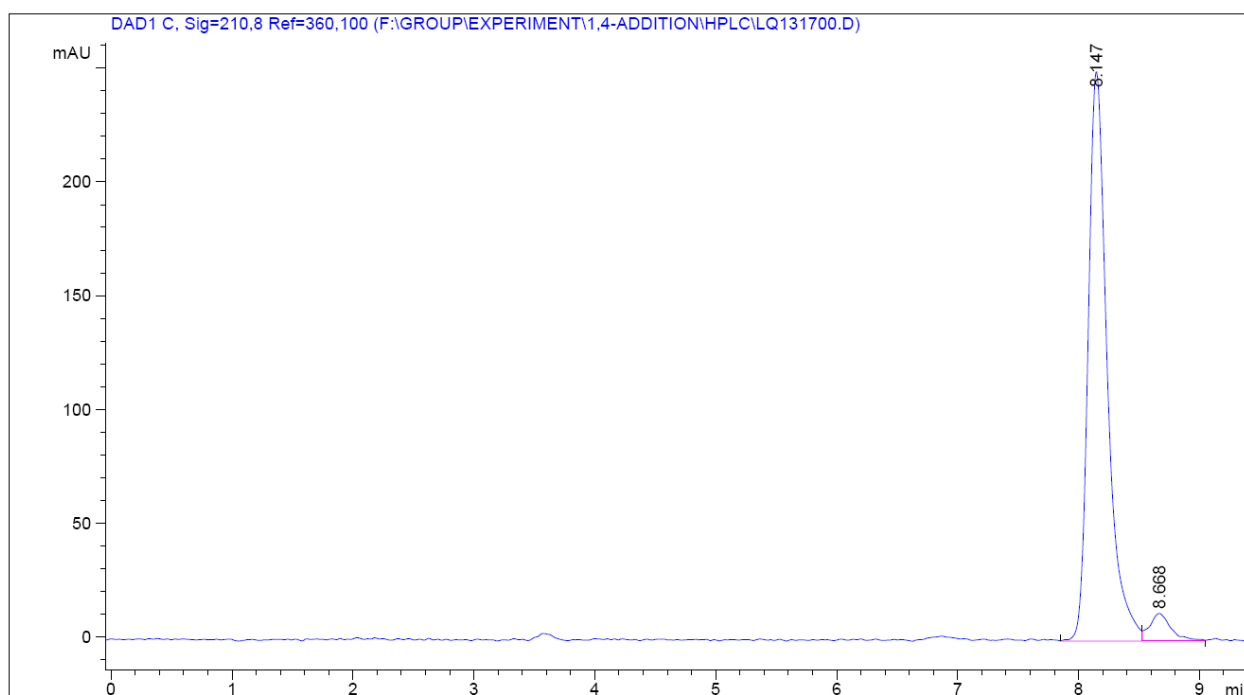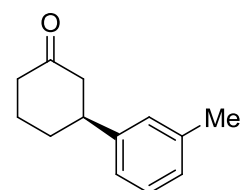
Racemic:

Table 2, entry 13

DAD1 C, Sig=210,8 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\LQ136000.D)

```
=====================================================================
                      Area Percent Report
=====================================================================

Sorted By             :       Signal
Multiplier            :       1.0000
Dilution              :       1.0000
Sample Amount         :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area        Height     Area
 #   [min]        [min]    [mAU*s]       [mAU]        %
----|-------|----|-------|----------|----------|--------|
 1   10.958 BV    0.2281 1.68056e4   1139.51489  49.6527
 2   11.774 VB    0.2426 1.70407e4   1088.54651  50.3473

Totals :                  3.38464e4   2228.06140
```
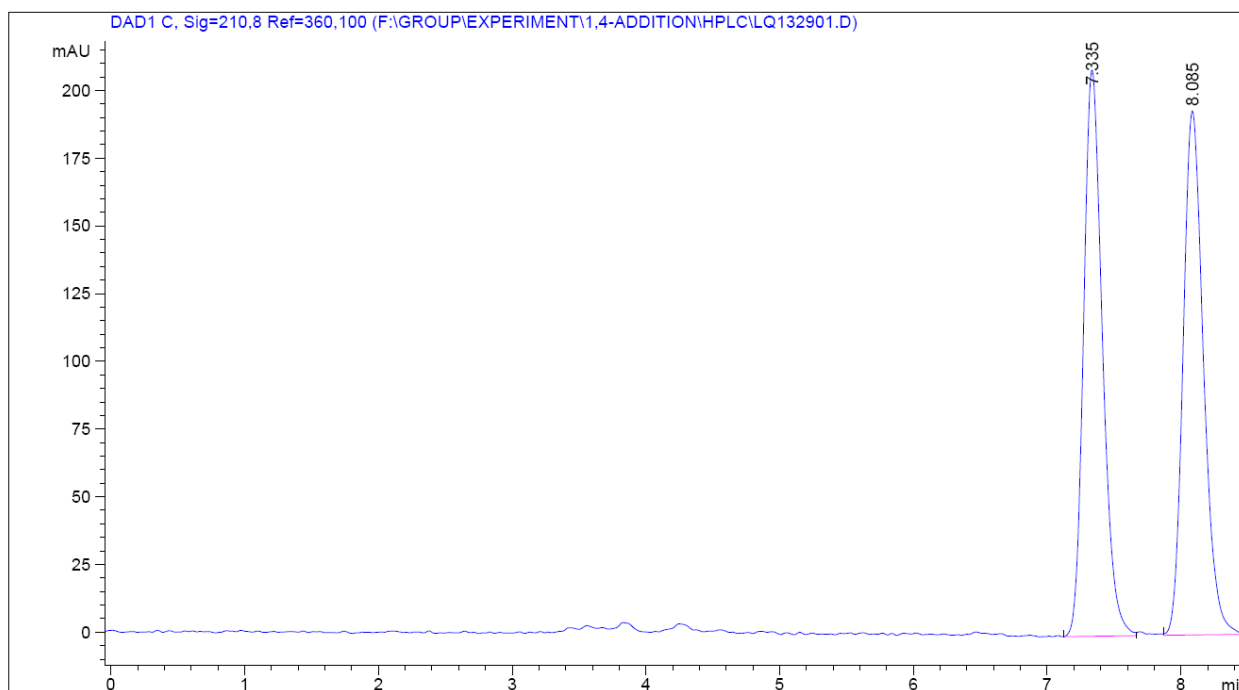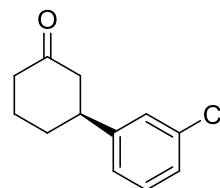
Chiral: **Table 2, entry 13**

```
====================================================================
                      Area Percent Report
====================================================================

Sorted By            :       Signal
Multiplier           :       1.0000
Dilution             :       1.0000
Sample Amount        :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height      Area
 #   [min]        [min]    [mAU*s]      [mAU]        %
----|-------|----|-------|----------|----------|--------|
  1  10.937 BV   0.2258 1.63678e4  1124.75464   94.1218
  2  11.748 VB   0.2310 1022.22516   65.17307    5.8782

Totals :                  1.73901e4  1189.92770
```
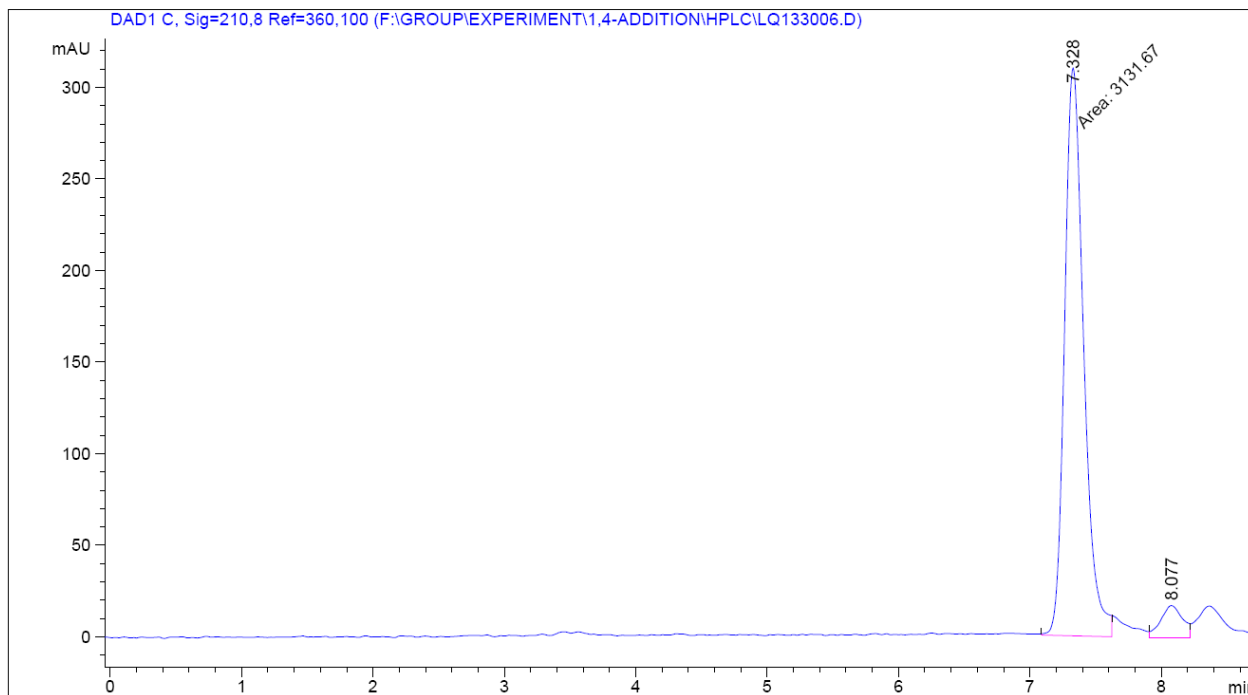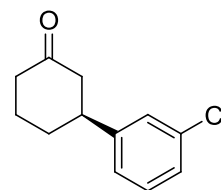
Racemic:

```
Acq. Operator   : SZB
Acq. Instrument : Instrument 1              Location :   -
Injection Date  : 2010-11-4 7:20:11
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-11-4 5:41:18  by SZB
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : AS-H, 0.5 ml/min, Hexane:iPrOH = 60:40
```
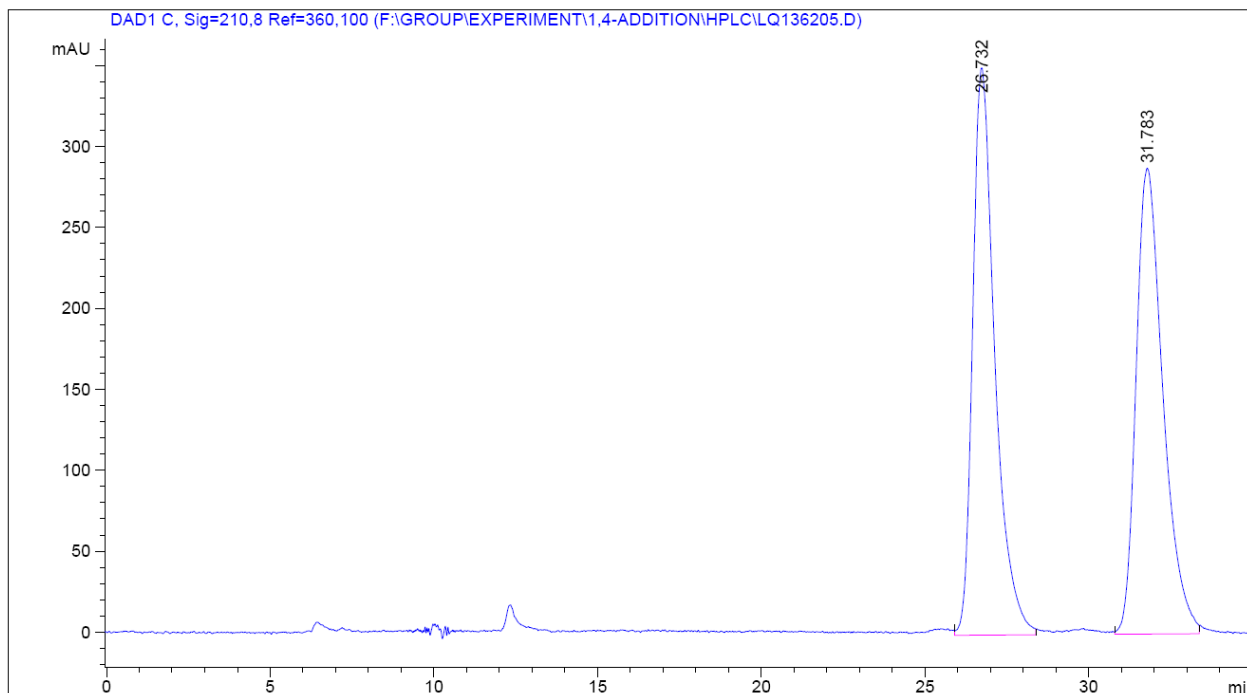


```
=====================================================================
                      Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area        Height     Area
  #   [min]        [min]   [mAU*s]       [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  26.732 VV   0.6215 1.64958e4    350.25821  50.0476
   2  31.783 VV   0.6858 1.64644e4    287.64984  49.9524

Totals :                  3.29601e4   637.90805
```

Chiral:

```
Acq. Operator    : SZB
Acq. Instrument  : Instrument 1                    Location :   -
Injection Date   : 2010-11-4 7:56:53
Acq. Method      : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed     : 2010-11-4 5:41:18  by SZB
                   (modified after loading)
Analysis Method  : C:\Chem32\1\METHODS\DEF_LC.M
Last changed     : 2004-4-7 0:10:12
Sample Info      : AS-H, 0.5 ml/min, Hexane:iPrOH = 60:40
```
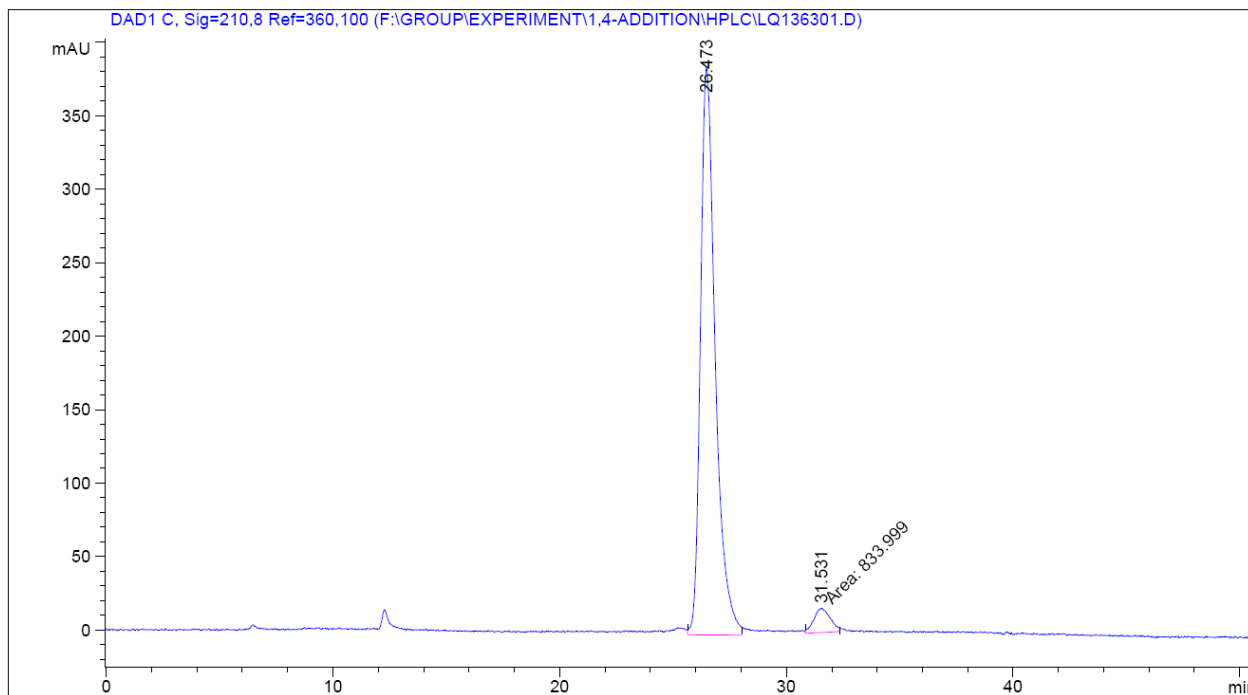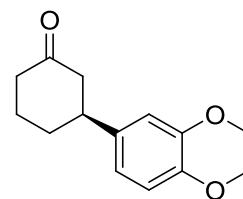


```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By            :        Signal
Multiplier           :        1.0000
Dilution             :        1.0000
Sample Amount        :        1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
  #   [min]        [min]   [mAU*s]      [mAU]       %
----|-------|----|-------|----------|----------|--------|
  1  26.473  VV   0.5836 1.75155e4   385.86618  95.4549
  2  31.531  MM   0.8369  833.99915   16.60858   4.5451

Totals :                  1.83495e4   402.47476
```
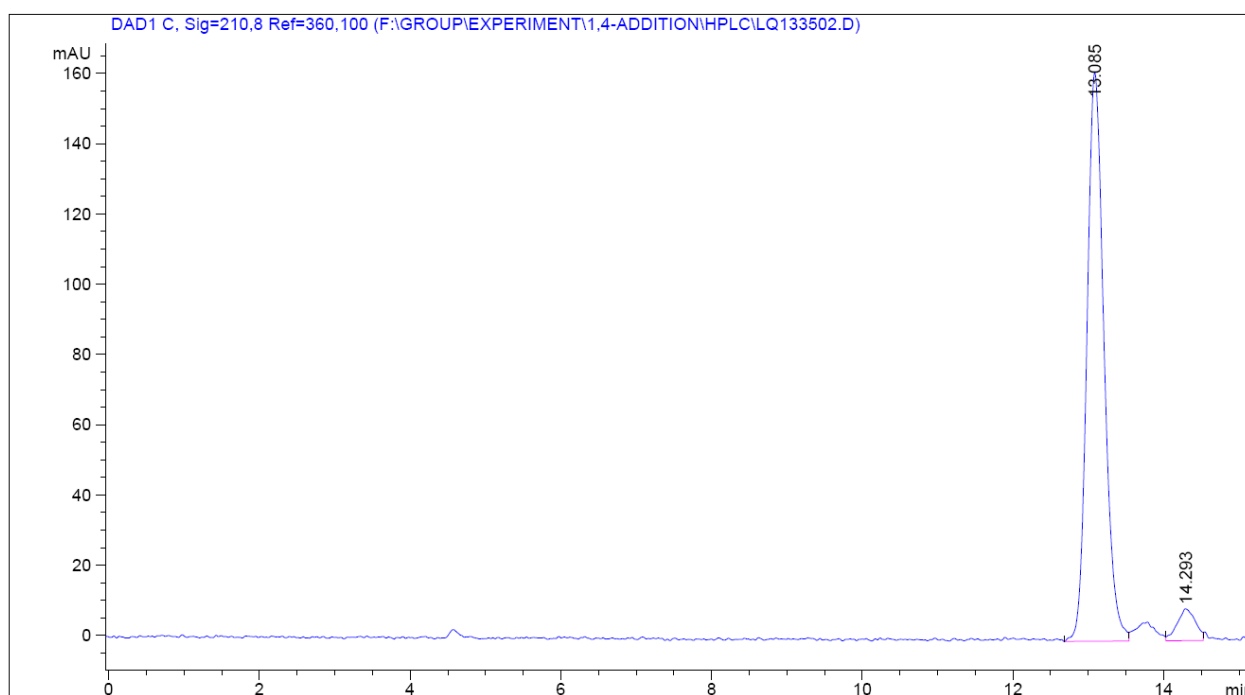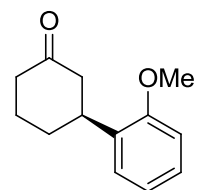
Racemic: **Table 2, entry 15**
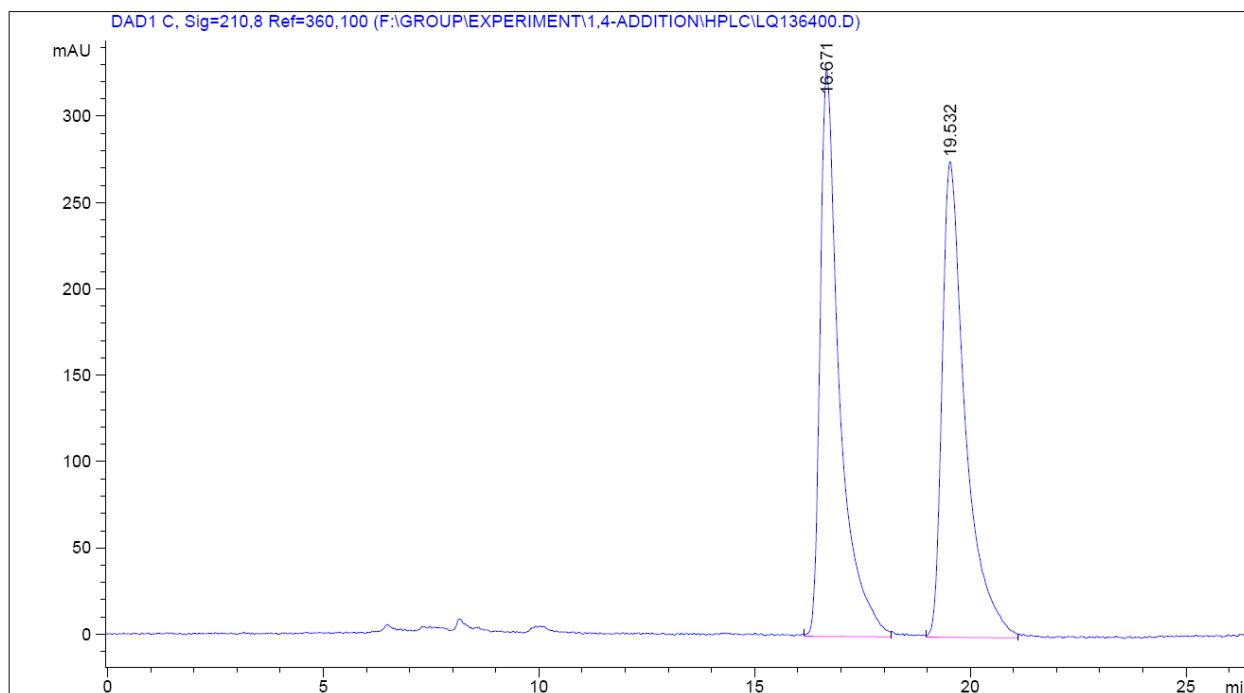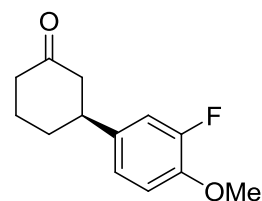
```
====================================================================
                       Area Percent Report
====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area        Height      Area
  #   [min]        [min]   [mAU*s]       [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1  16.671 VB   0.4469 1.02687e4   328.27240  49.9691
   2  19.532 VV   0.5355 1.02814e4   275.41440  50.0309

Totals :                  2.05500e4   603.68680
```

Chiral:

Table 2, entry 15

DAD1 C, Sig=210,8 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\LQ136501.D)

```
=====================================================================
                    Area Percent Report
=====================================================================

Sorted By          :       Signal
Multiplier         :       1.0000
Dilution           :       1.0000
Sample Amount      :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height      Area
  #   [min]        [min]   [mAU*s]      [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1  16.594 VV    0.4273 9233.49219  313.75320  95.0027
   2  19.455 MM    0.4907  485.69437   16.49713   4.9973

Totals :                  9719.18655  330.25033
```

Racemic:

Table 2, entry 16

DAD1 C, Sig=210,8 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\LQ137101.D)


```
=====================================================================
                       Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area        Height     Area
  #   [min]        [min]   [mAU*s]       [mAU]       %
----|-------|----|-------|----------|----------|--------|
  1  15.046 VV    0.4282 1.51109e4    524.42950  49.9148
  2  21.570 VV    0.5186 1.51625e4    360.54782  50.0852

Totals :                  3.02734e4   884.97733
```
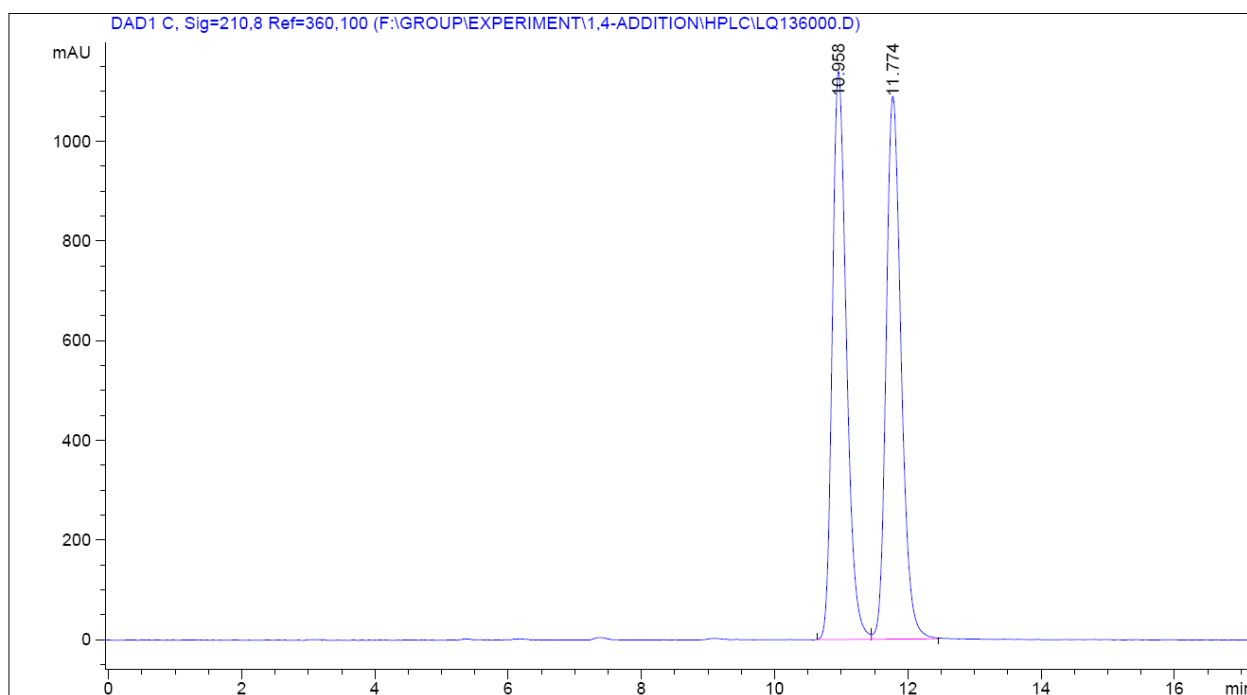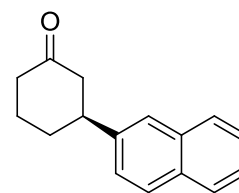
Chiral:

```
Acq. Operator   : SZB
Acq. Instrument : Instrument 1              Location :   -
Injection Date  : 2010-11-5 12:16:18
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-11-5 11:49:22  by SZB
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : AD-H, 0.5 ml/min, Hexane:iPrOH = 60:40
```
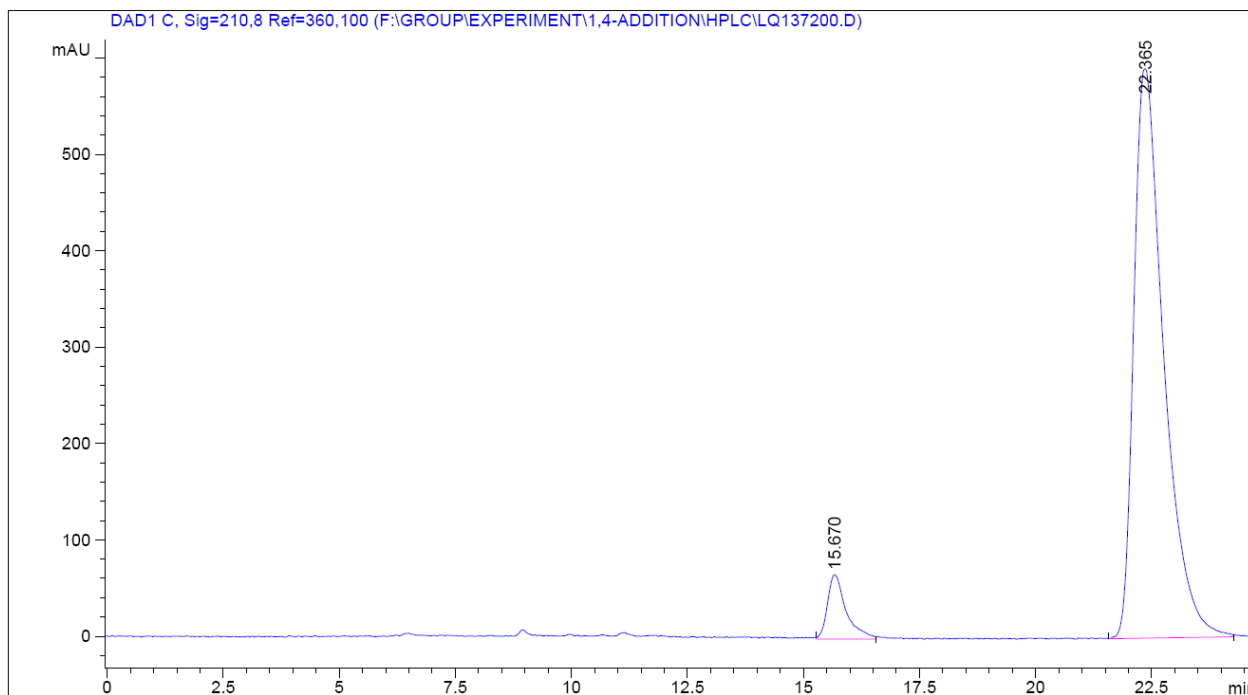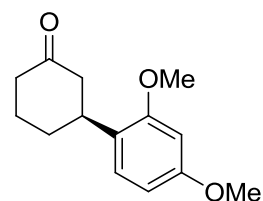


```
=====================================================================
                      Area Percent Report
=====================================================================

Sorted By             :       Signal
Multiplier            :       1.0000
Dilution              :       1.0000
Sample Amount         :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area        Height      Area
  #   [min]        [min]    [mAU*s]       [mAU]        %
----|-------|----|-------|----------|----------|--------|
  1  15.670  VV   0.3849 1866.36145   67.02787   6.5784
  2  22.365  VB   0.5753 2.65047e4    590.87689  93.4216

Totals :                  2.83710e4    657.90476
```
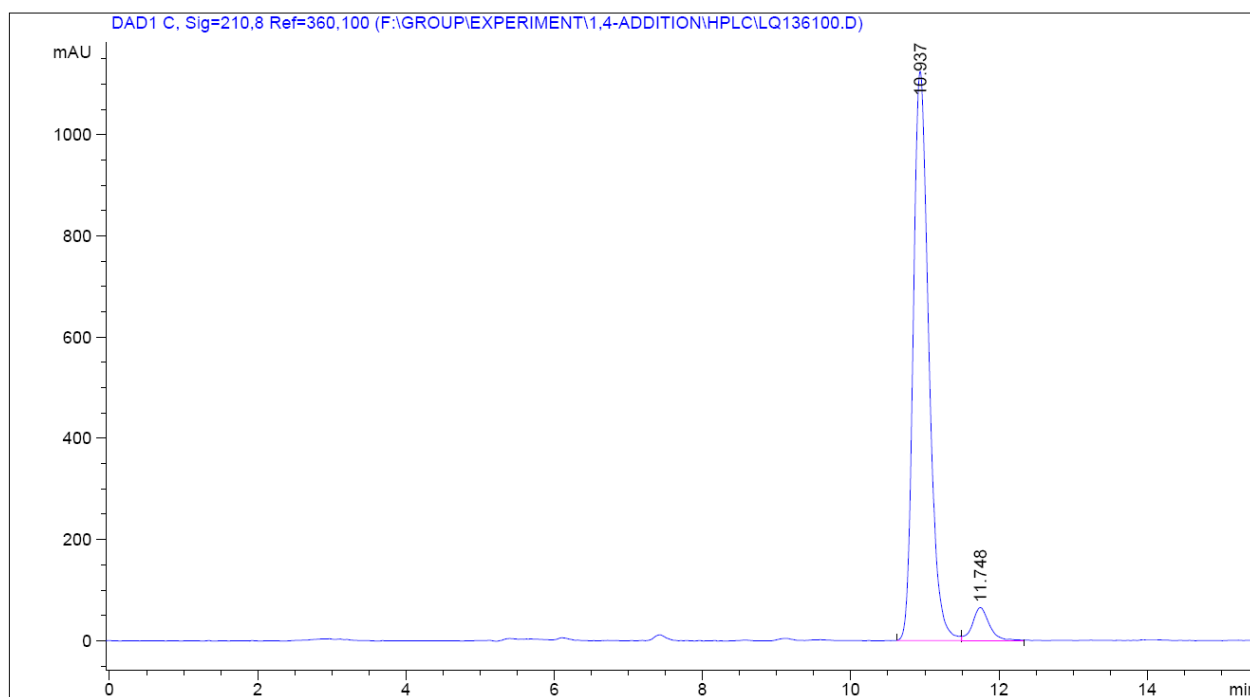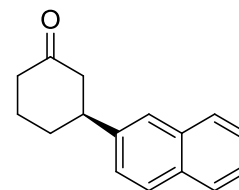
Table 2, entry 17
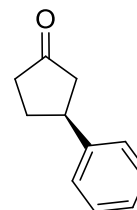
Racemic:

LQ1241-1.DATA - Prostar 335 Absorbance Analog Channel 1 EL06099083
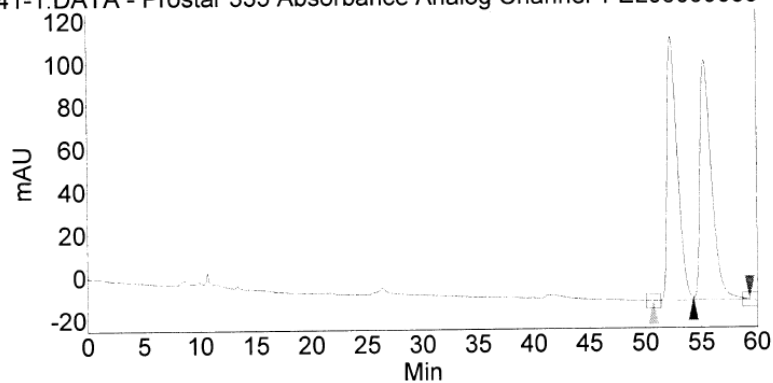
Index	Time [Min]	Area % [%]
1	52.36	49.196
2	55.31	50.804
Total		100.000

Chiral:

LQ1242-1.DATA - Prostar 335 Absorbance Analog Channel 1 EL06099083
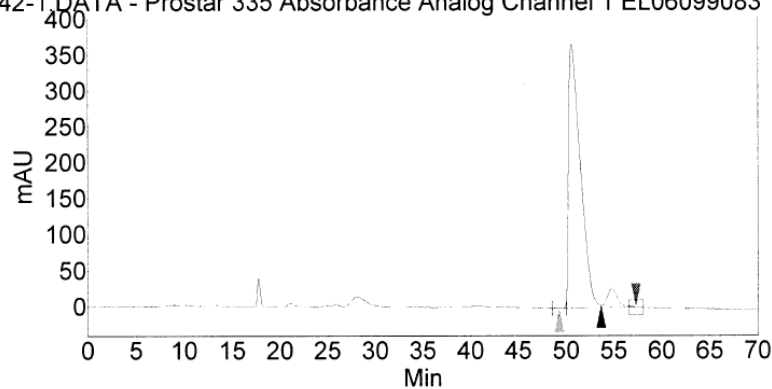
Index	Time [Min]	Area % [%]
1	50.53	94.163
2	54.79	5.837
Total		100.000

Racemic: **Table 2, entry 18**

```
Acq. Operator   : LQ
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 2010-10-20 5:26:25
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-10-20 5:10:44  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : OJ-H, 0.7 ml/min, Hexane:iPrOH = 98:2
```
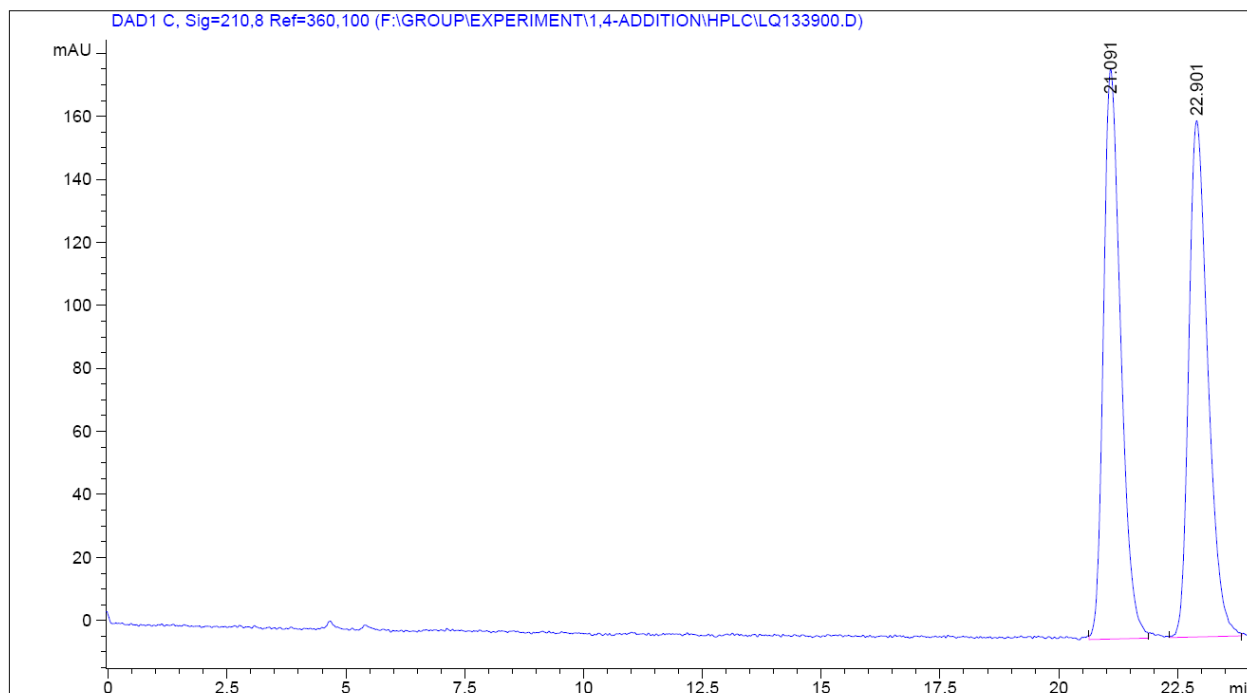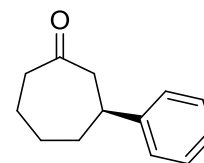


```
=====================================================================
                      Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
 #   [min]        [min]    [mAU*s]      [mAU]       %
----|-------|----|-------|----------|----------|--------|
  1  21.091 VV   0.3692 4578.00146  180.93985  50.0466
  2  22.901 VV   0.4154 4569.48242  163.83353  49.9534

Totals :                  9147.48389  344.77338
```
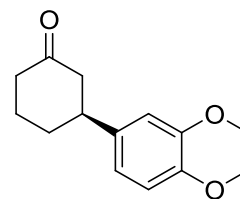
Chiral: **Table 2, entry 18**

```
Acq. Operator   : LQ
Acq. Instrument : Instrument 1                     Location :    -
Injection Date  : 2010-10-20 5:52:22
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-10-20 5:10:44  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : OJ-H, 0.7 ml/min, Hexane:iPrOH = 98:2
```



```
====================================================================
                      Area Percent Report
====================================================================

Sorted By            :       Signal
Multiplier           :       1.0000
Dilution             :       1.0000
Sample Amount        :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
  #   [min]        [min]   [mAU*s]      [mAU]        %
----|-------|----|-------|----------|----------|--------|
  1  20.935 VB    0.3919 8529.62988  327.46295  92.5124
  2  22.856 BB    0.3558  690.35907   24.12905   7.4876

Totals :                  9219.98895  351.59200
```

Racemic:

Table 2, entry 19

```
=======================================================================
                         Area Percent Report
=======================================================================

Sorted By            :      Signal
Multiplier           :      1.0000
Dilution             :      1.0000
Sample Amount        :      1.00000  [ng/ul]    (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area        Height      Area
  #   [min]        [min]    [mAU*s]      [mAU]         %
----|-------|----|-------|----------|----------|--------|
   1  27.202 VB   0.6181 1.92013e4   370.79807   49.9134
   2  31.792 VV   0.7477 1.92679e4   308.90521   50.0866

Totals :                  3.84692e4   679.70328
```
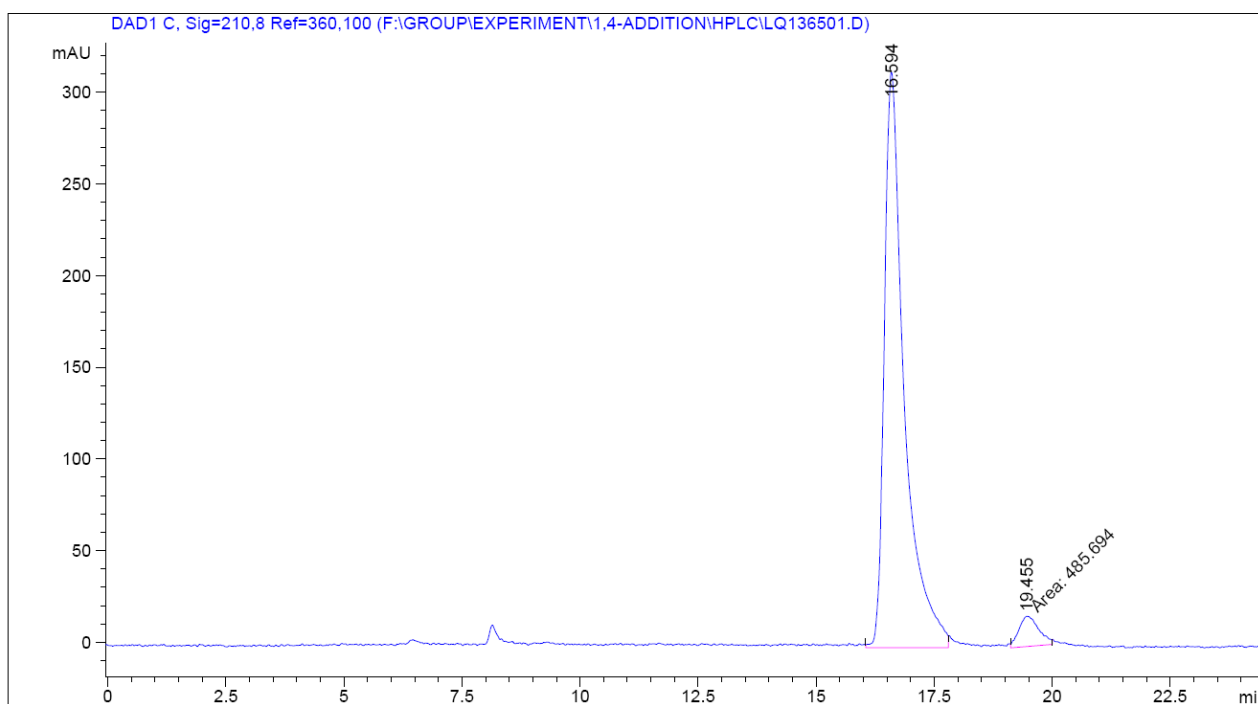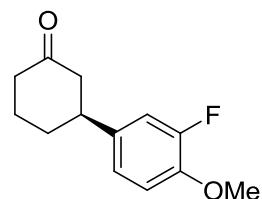
Chiral:

```
Acq. Operator   : LQ
Acq. Instrument : Instrument 1              Location :   -
Injection Date  : 2010-10-29 9:56:18
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-10-29 4:30:44  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : AS-H, 0.6 ml/min, Hexane:iPrOH = 65:35
```



```
=====================================================================
                      Area Percent Report
=====================================================================

Sorted By             :       Signal
Multiplier            :       1.0000
Dilution              :       1.0000
Sample Amount         :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
  #   [min]        [min]   [mAU*s]      [mAU]       %
----|-------|----|-------|----------|----------|--------|
  1  27.040 VV    0.5105 1641.01953   38.40594   4.0887
  2  31.179 MM    1.0597 3.84943e4   605.43646  95.9113

Totals :                  4.01353e4   643.84241
```

5. Nonlinear Effect Study of the Standard Reaction

   The reactions were done following the same procedures as stated above in the representative procedures using chiral diene ligand **1a** in different ee's, which were determined by HPLC. After the completion of the reaction, the product was separated and purified by column chromatography, and the ee's of these products were also determined by HPLC analysis. The ee's of the chiral diene ligands and corresponding products were shown in the table below.

ee of ligand	13%	27%	41%	56%	69%	81%	94%	100%
ee of product	9%	21%	32%	48%	60%	70%	91%	97%

These date showd there was no nonliner effect in this asymmetric addition reaction:

ee of product (%)

$y = 1.008x - 7.157$

$R^2 = 0.990$

ee of ligand (%)
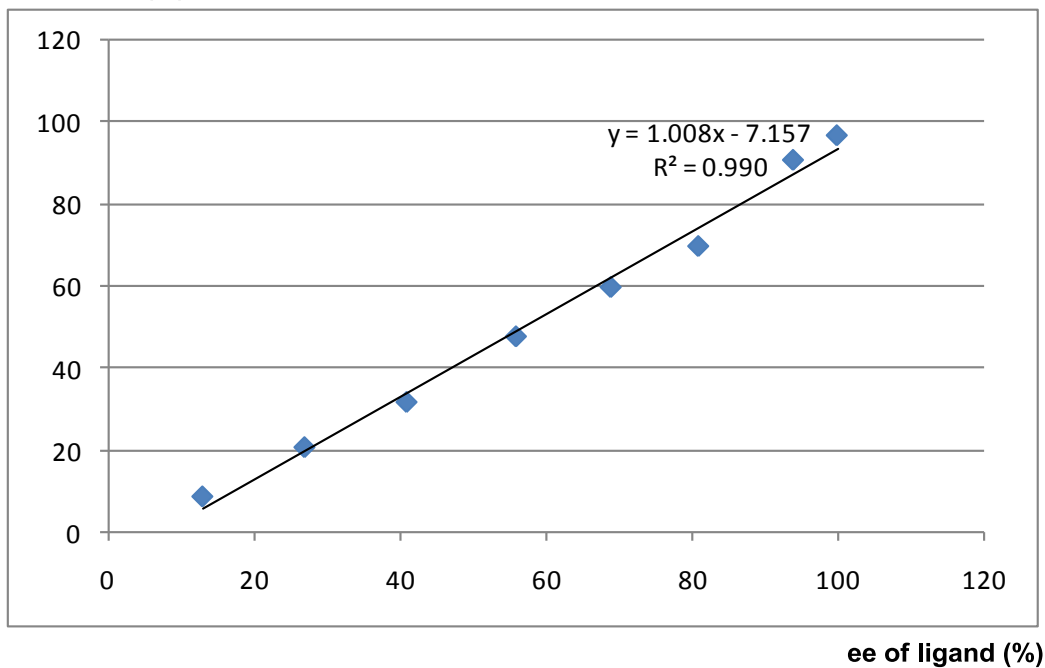
1. The first spot

(1) ee of the ligand

```
Acq. Operator   : LQ
Acq. Instrument : Instrument 1              Location :    -
Injection Date  : 2010-11-20 5:52:05
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-11-20 0:13:44  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : OD-H, 1.0 ml/min, Hexane:iPrOH = 99:1
```



```
=====================================================================
                       Area Percent Report
=====================================================================

Sorted By            :       Signal
Multiplier           :       1.0000
Dilution             :       1.0000
Sample Amount        :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area        Height      Area
  #   [min]        [min]   [mAU*s]       [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1  10.701 BV   0.3102 2482.56958  123.66018  56.6255
   2  12.617 VB   0.3928 1901.62329   74.75568  43.3745

Totals :                  4384.19287  198.41586
```

(2) ee of the product

DAD1 C, Sig=210,8 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\DZ056003.D)

```
=====================================================================
                      Area Percent Report
=====================================================================

Sorted By           :       Signal
Multiplier          :       1.0000
Dilution            :       1.0000
Sample Amount       :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
  #   [min]        [min]   [mAU*s]      [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1   7.473 VB   0.1472 1205.81653  124.31672  54.6353
   2   8.826 BB   0.1665 1001.21185   90.88689  45.3647

Totals :                 2207.02838  215.20361
```

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2. The second spot

(1) ee of the ligand

Acq. Operator   : LQ
Acq. Instrument : Instrument 1   Location :   -
Injection Date : 2010-11-20 5:35:05
Acq. Method   : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed   : 2010-11-20 0:13:44   by LQ
                   (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed   : 2004-4-7 0:10:12
Sample Info   : OD-H, 1.0 ml/min, Hexane:iPrOH = 99:1

```
=====================================================================
                      Area Percent Report
=====================================================================

Sorted By          :      Signal
Multiplier         :      1.0000
Dilution           :      1.0000
Sample Amount      :      1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area        Height     Area
  #   [min]        [min]   [mAU*s]       [mAU]       %
----|-------|----|-------|----------|----------|--------|
  1  10.708 BV   0.3141 2933.67236  146.17662  63.6609
  2  12.633 VB   0.3922 1674.60449   66.40054  36.3391

Totals :                 4608.27686  212.57716
```
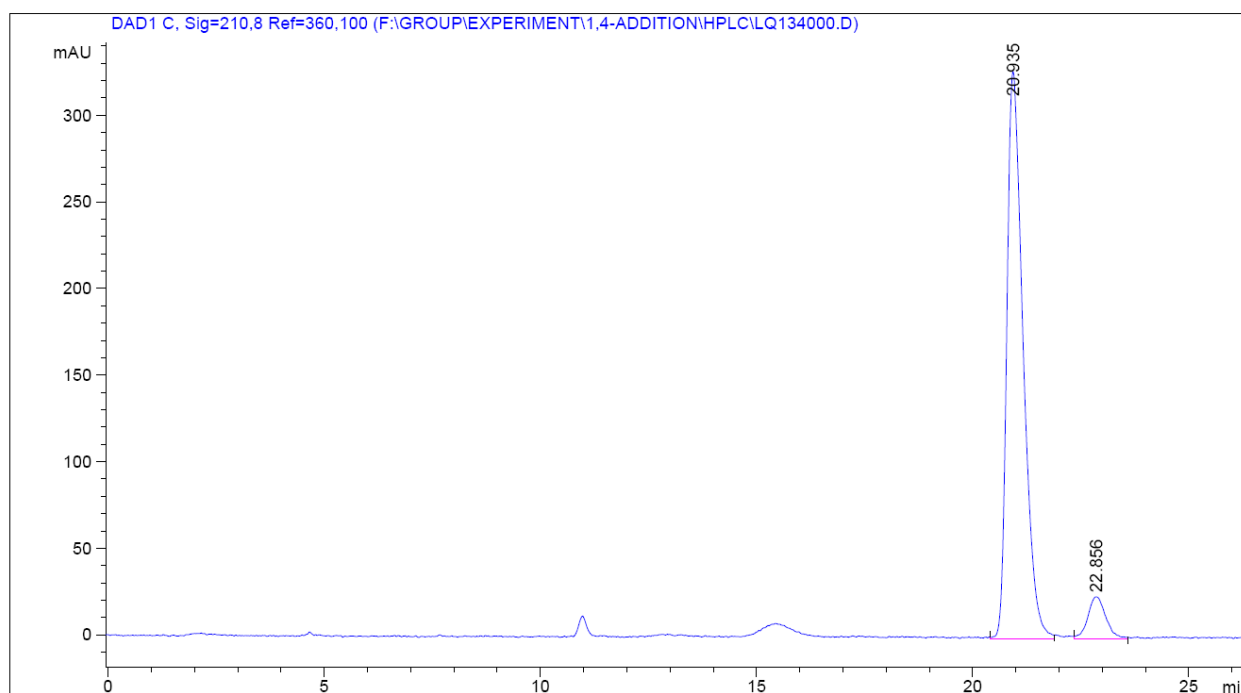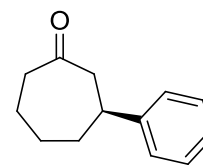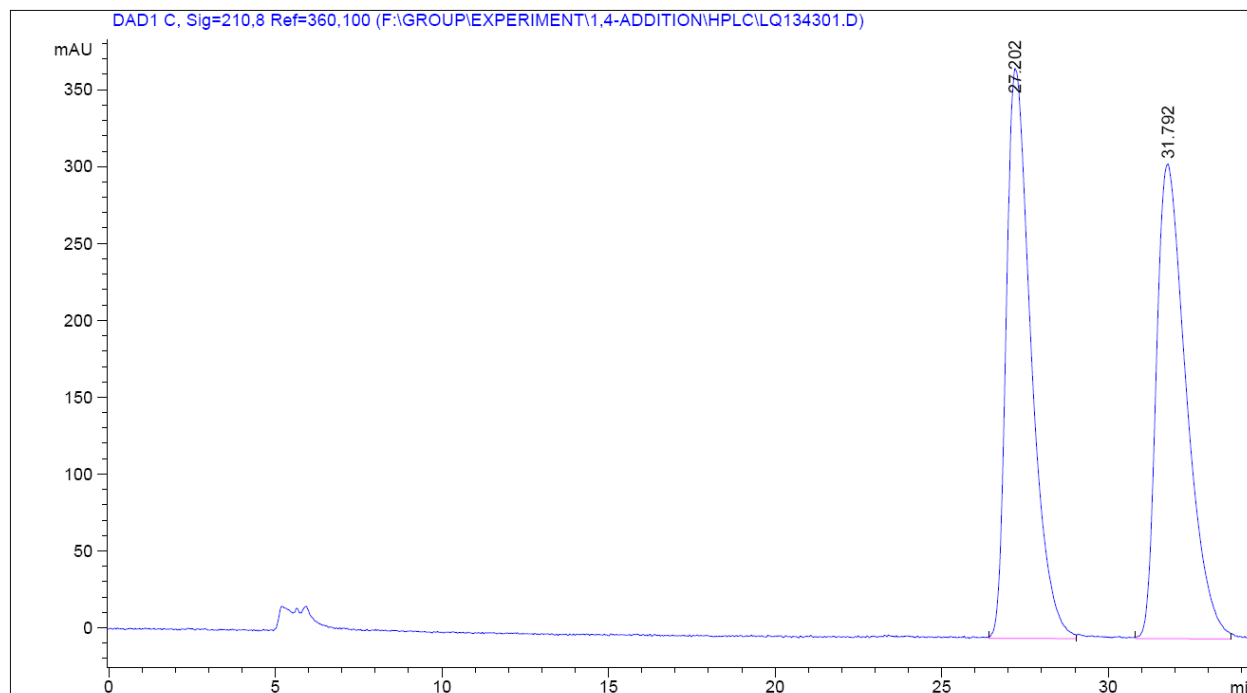
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(2) ee of the product

```
Acq. Operator   : LQ
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 2010-11-23 0:32:32
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-11-22 23:35:35  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : AD-H, 1.0 ml/min, Hexane:iPrOH = 97:3
```

DAD1 C, Sig=210,8 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\DZ057000.D)

```
=====================================================================
                      Area Percent Report
=====================================================================

Sorted By             :        Signal
Multiplier            :        1.0000
Dilution              :        1.0000
Sample Amount         :        1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
 #    [min]        [min]    [mAU*s]      [mAU]       %
----|-------|----|-------|----------|----------|--------|
  1   7.551 VB   0.1485  1384.79956  141.15367  60.5363
  2   8.912 VV   0.1735   902.75458   80.10446  39.4637

Totals :                  2287.55414  221.25813
```
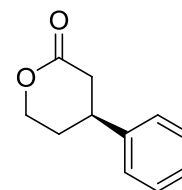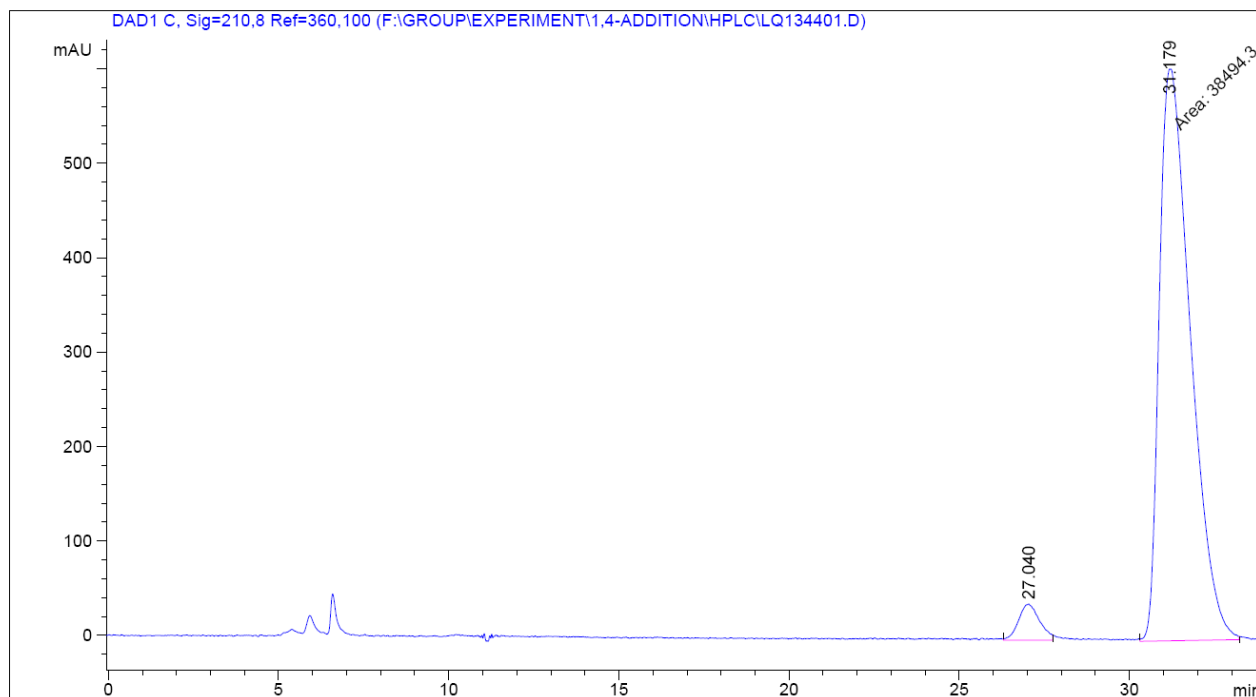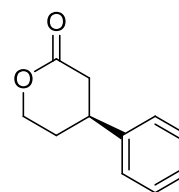
3. The third spot

(1) ee of the ligand

Acq. Operator   : LQ
Acq. Instrument : Instrument 1   Location :   -
Injection Date   : 2010-11-20 4:39:12
Acq. Method   : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed   : 2010-11-20 0:13:44   by LQ
                   (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed   : 2004-4-7 0:10:12
Sample Info   : OD-H, 1.0 ml/min, Hexane:iPrOH = 99:1

DAD1 B, Sig=254,16 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\LQ-LINE4.D)

```
=====================================================================
                     Area Percent Report
=====================================================================

Sorted By            :       Signal
Multiplier           :       1.0000
Dilution             :       1.0000
Sample Amount        :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area        Height      Area
  #   [min]        [min]    [mAU*s]       [mAU]        %
----|-------|----|-------|----------|----------|--------|
  1  10.749 BV   0.3157 4109.88330  200.06824  70.4298
  2  12.690 VB   0.3798 1725.54504   67.14547  29.5702

Totals :                 5835.42834  267.21371
```
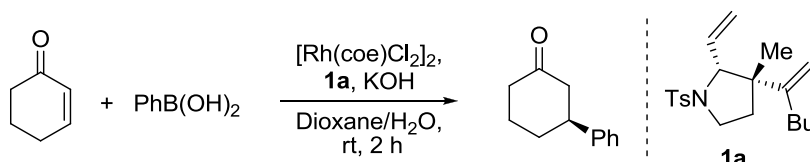
(2) ee of the product

```
Acq. Operator   : LQ
Acq. Instrument : Instrument 1                 Location :   -
Injection Date  : 2010-11-23 1:05:36
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-11-22 23:35:35  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : AD-H, 1.0 ml/min, Hexane:iPrOH = 97:3
```

DAD1 C, Sig=210,8 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\LQ140100.D)

```
========================================================================
                        Area Percent Report
========================================================================

Sorted By             :       Signal
Multiplier            :       1.0000
Dilution              :       1.0000
Sample Amount         :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area        Height      Area
  #    [min]       [min]    [mAU*s]      [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1   7.878 VV   0.1570 1888.14465  185.13675  65.8897
   2   9.299 VB   0.1849  977.47119   80.94292  34.1103

Totals :                  2865.61584  266.07967
```

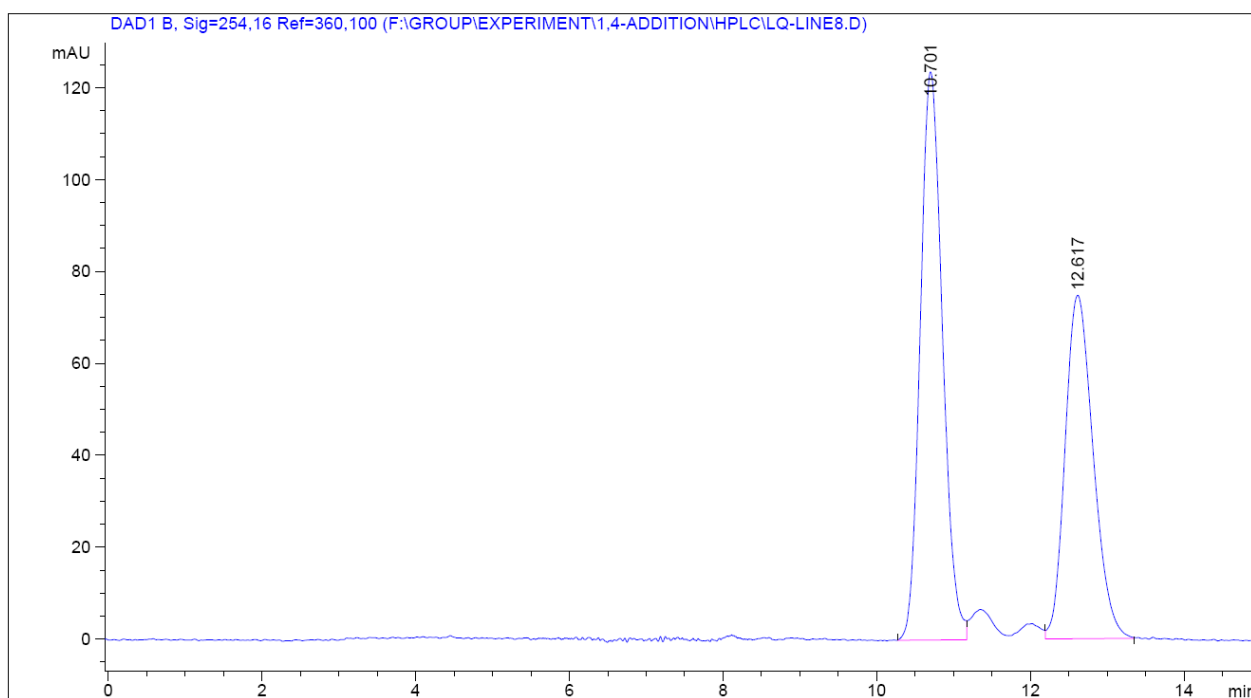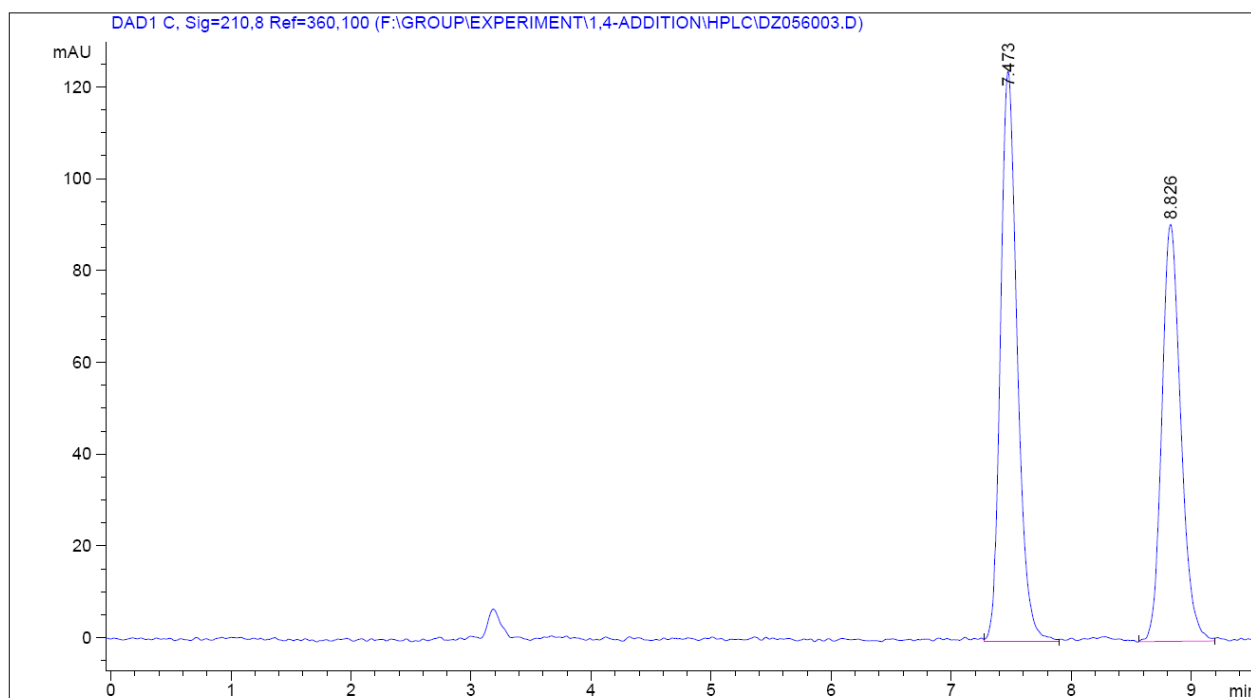4. The fourth spot

(1) ee of the ligand

```
Acq. Operator   : LQ
Acq. Instrument : Instrument 1                    Location :    -
Injection Date  : 2010-11-20 4:18:54
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-11-20 0:13:44  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : OD-H, 1.0 ml/min, Hexane:iPrOH = 99:1
```


DAD1 B, Sig=254,16 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\LQ-LINE3.D)

```
=====================================================================
                       Area Percent Report
=====================================================================

Sorted By             :        Signal
Multiplier            :        1.0000
Dilution              :        1.0000
Sample Amount         :        1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area        Height     Area
  #   [min]        [min]    [mAU*s]       [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.776 BV   0.3228 3442.50244  164.03116  77.9587
   2  12.737 VV   0.3753  973.29889   37.68314  22.0413

Totals :                  4415.80133  201.71430
```
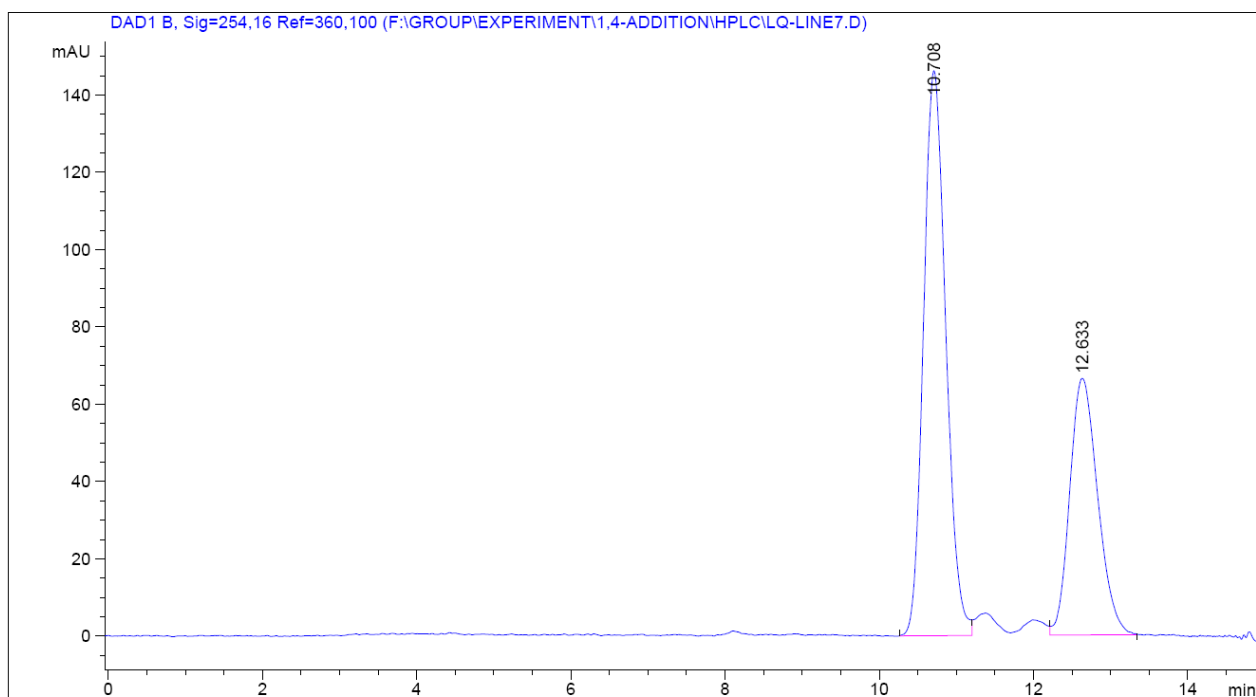
(2) ee of the product

```
Acq. Operator   : LQ
Acq. Instrument : Instrument 1                  Location :   -
Injection Date  : 2010-11-23 1:30:33
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-11-22 23:35:35  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : AD-H, 1.0 ml/min, Hexane:iPrOH = 97:3
```

DAD1 C, Sig=210,8 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\LQ140200.D)

```
=====================================================================
                       Area Percent Report
=====================================================================

Sorted By             :        Signal
Multiplier            :        1.0000
Dilution              :        1.0000
Sample Amount         :        1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area        Height      Area
  #   [min]        [min]    [mAU*s]      [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1   8.425 VV   0.1646 1608.34216  148.23921  74.1814
   2   9.946 VB   0.1939  559.77911   45.39549  25.8186

Totals :                  2168.12128  193.63470
```
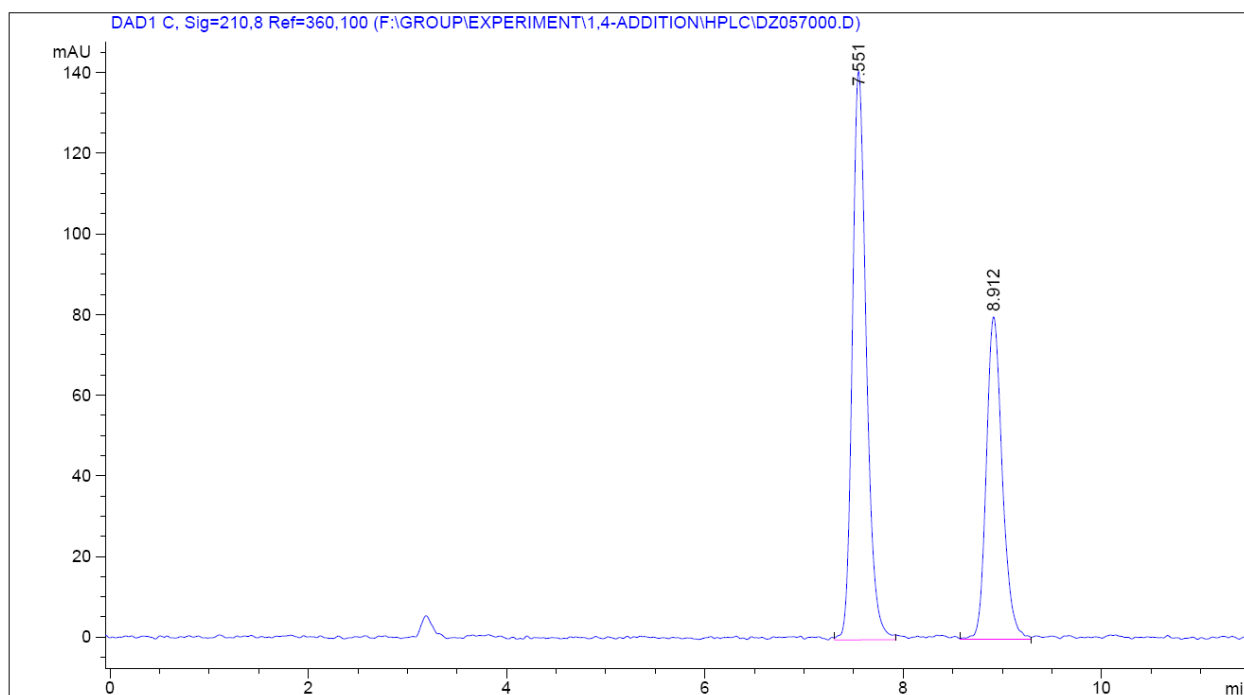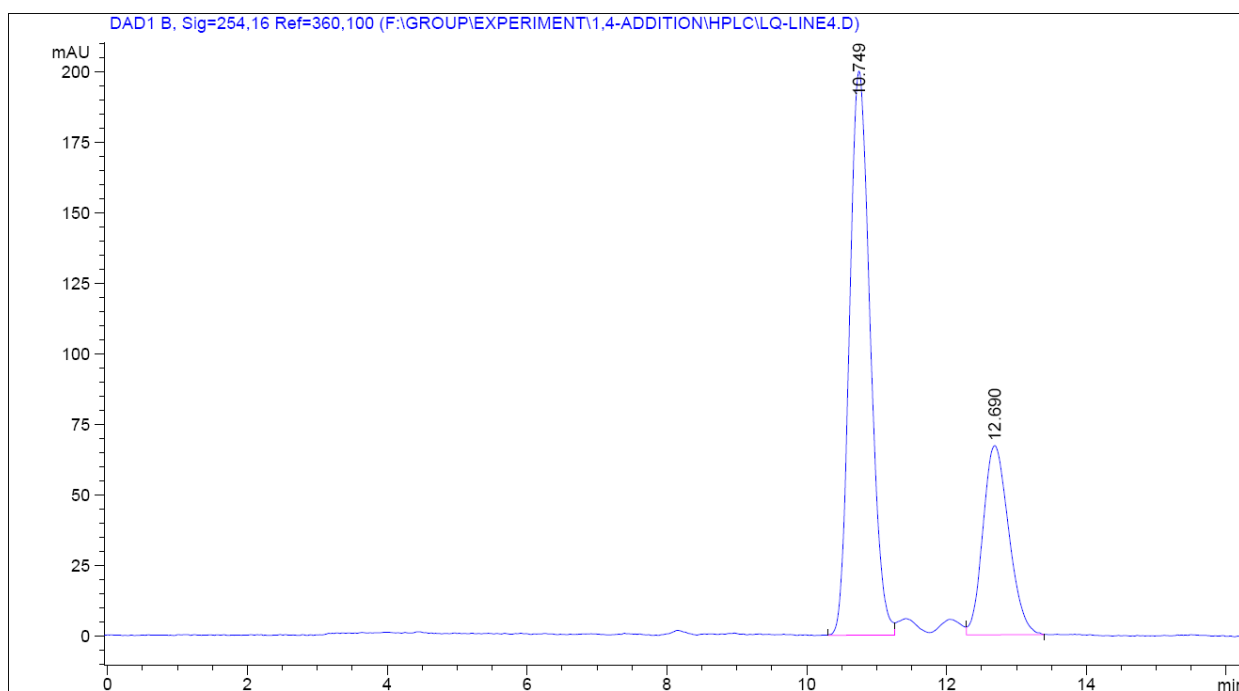
5. The fifth spot

(1) ee of the ligand

Acq. Operator   : LQ
Acq. Instrument : Instrument 1   Location :   -
Injection Date : 2010-11-20 5:16:51
Acq. Method   : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed   : 2010-11-20 0:13:44 by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed   : 2004-4-7 0:10:12
Sample Info   : OD-H, 1.0 ml/min, Hexane:iPrOH = 99:1

```
=====================================================================
                       Area Percent Report
=====================================================================

Sorted By          :        Signal
Multiplier         :        1.0000
Dilution           :        1.0000
Sample Amount      :        1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area        Height     Area
  #   [min]        [min]    [mAU*s]      [mAU]        %
----|-------|----|-------|----------|----------|--------|
  1  10.672 BB   0.3224 6781.15527  329.07346  84.6817
  2  12.659 VB   0.3863 1226.66516   48.30281  15.3183

Totals :                 8007.82043  377.37626
```
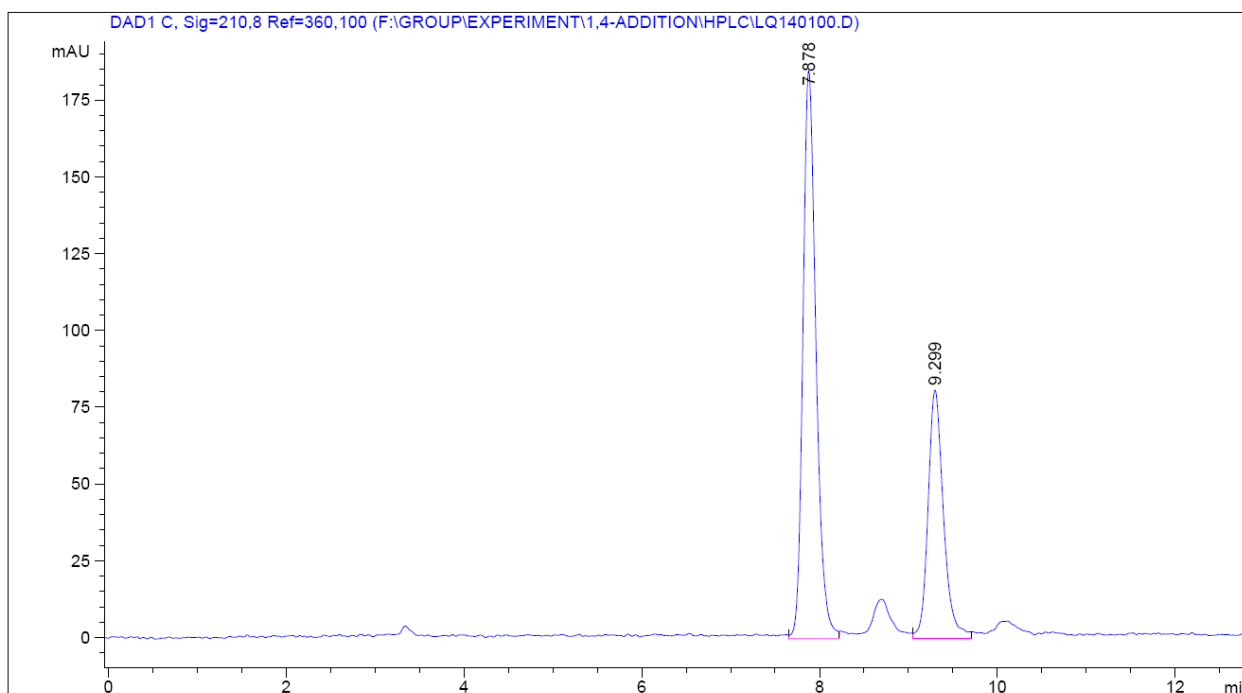
(2) ee of the product

```
Acq. Operator    : LQ
Acq. Instrument : Instrument 1                    Location :    -
Injection Date   : 2010-11-23 3:20:32
Acq. Method      : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed     : 2010-11-23 2:41:17  by LQ
                   (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed     : 2004-4-7 0:10:12
Sample Info      : AD-H, 1.0 ml/min, Hexane:iPrOH = 97:3
```

DAD1 C, Sig=210,8 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\LQ140301.D)

```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :       Signal
Multiplier            :       1.0000
Dilution              :       1.0000
Sample Amount         :       1.00000  [ng/ul]    (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area        Height      Area
  #   [min]        [min]   [mAU*s]       [mAU]        %
----|-------|----|-------|----------|----------|--------|
   1   8.967 VV    0.1793 2822.71411  243.45937  80.0250
   2  10.587 VV    0.1968  704.57581   53.78538  19.9750

Totals :                  3527.28992  297.24475
```
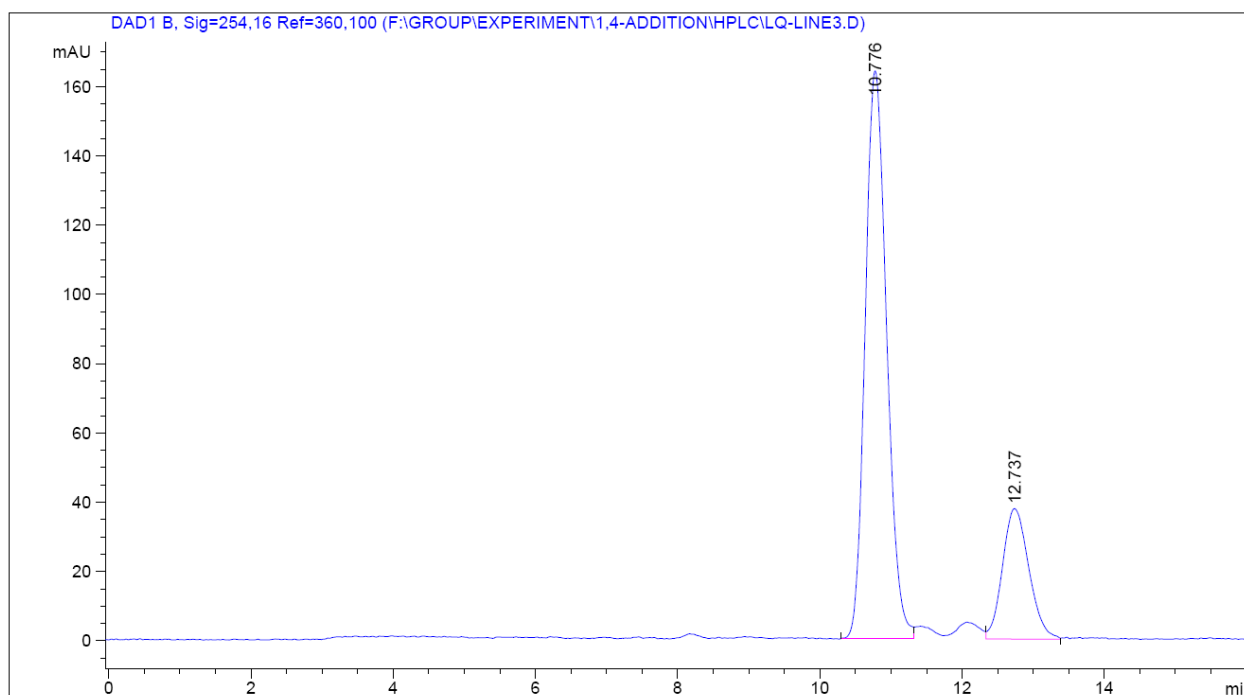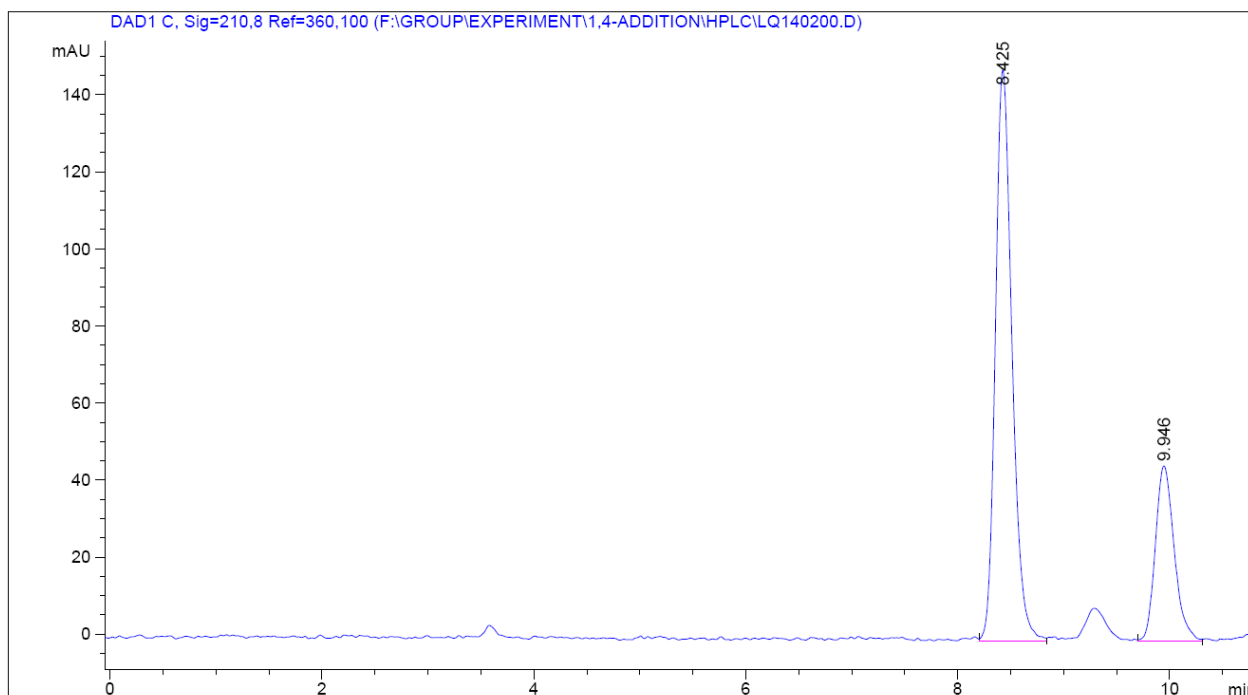
S70

6. The sixth spot

(1) ee of the ligand

```
Acq. Operator   : LQ
Acq. Instrument : Instrument 1                    Location :    -
Injection Date  : 2010-11-20 3:34:26
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-11-20 0:13:44  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : OD-H, 1.0 ml/min, Hexane:iPrOH = 99:1
```


DAD1 B, Sig=254,16 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\LQ-LINE1.D)

```
=====================================================================
                    Area Percent Report
=====================================================================

Sorted By         :       Signal
Multiplier        :       1.0000
Dilution          :       1.0000
Sample Amount     :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.640 BB   0.3146 3565.75757  177.30003  90.3805
   2  12.562 VB   0.3460  379.51337   15.70200   9.6195

Totals :                 3945.27094  193.00203
```
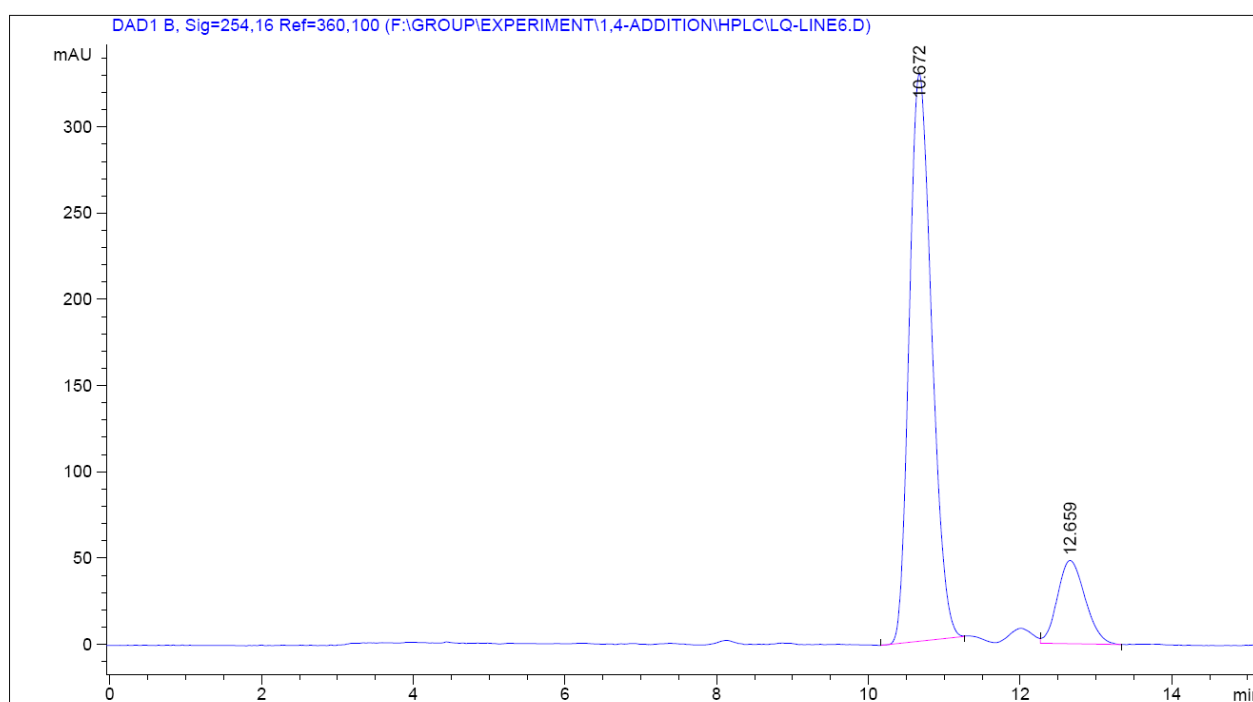
(2) ee of the product

```
Acq. Operator   : LQ
Acq. Instrument : Instrument 1                    Location :    -
Injection Date  : 2010-11-23 3:45:23
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-11-23 2:41:17  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : AD-H, 1.0 ml/min, Hexane:iPrOH = 97:3
```


DAD1 C, Sig=210,8 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\LQ140400.D)

```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By            :        Signal
Multiplier           :        1.0000
Dilution             :        1.0000
Sample Amount        :        1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
 #   [min]        [min]    [mAU*s]      [mAU]       %
----|-------|----|-------|----------|----------|--------|
  1    9.641 VB   0.1901 3192.45020  254.94325  85.0033
  2   11.385 VB   0.2223  563.22815   38.58564  14.9967

Totals :                 3755.67834  293.52889
```
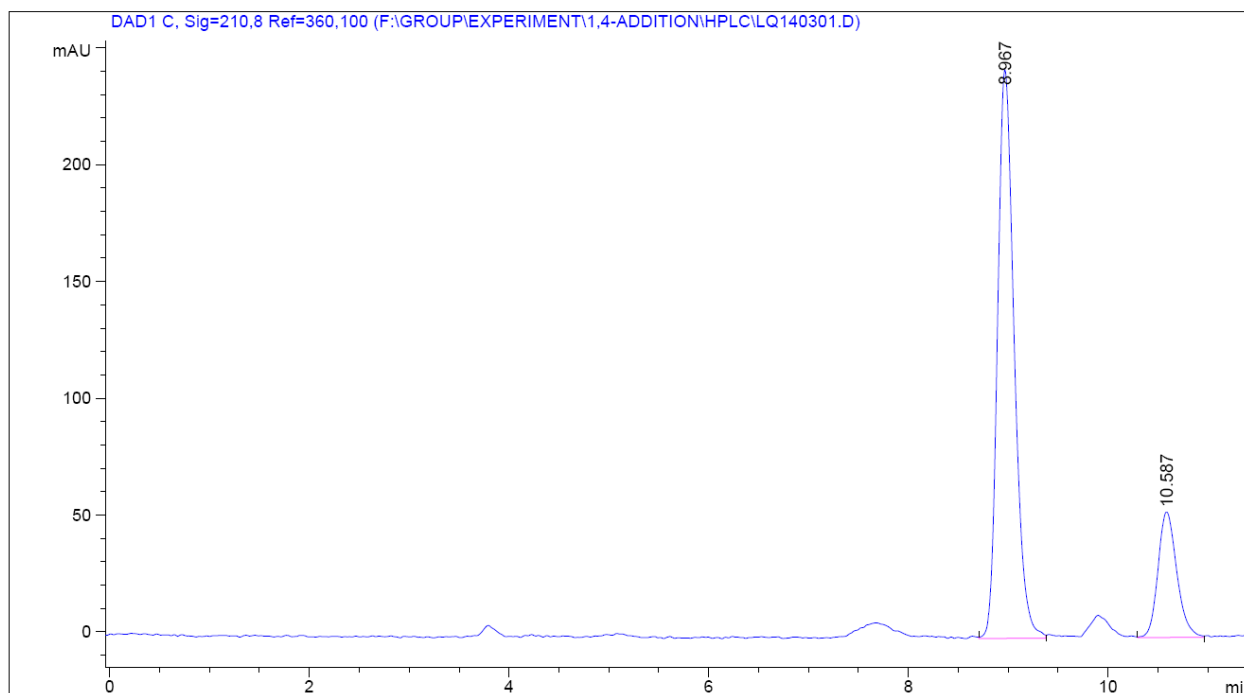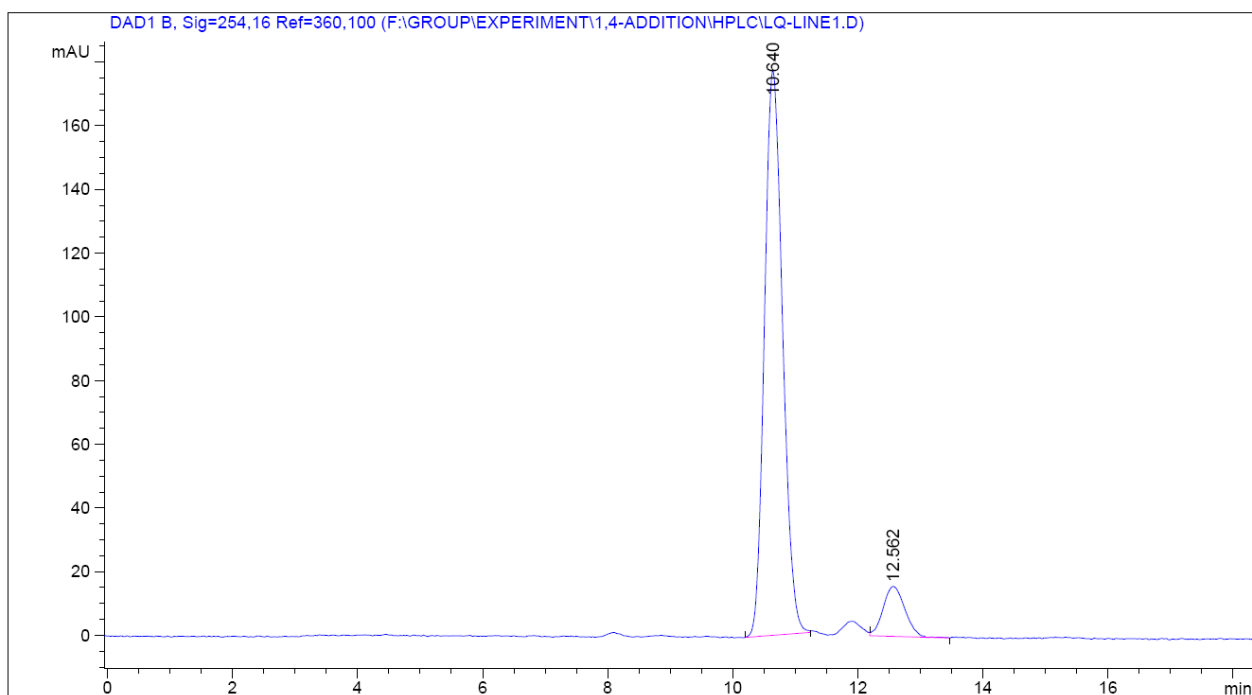
7. The seventh spot

(1) ee of the ligand

```
Acq. Operator   : LQ
Acq. Instrument : Instrument 1                    Location :    -
Injection Date  : 2010-7-21 4:24:59
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-7-21 3:50:17  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : OD-H, 1.0 mL/min, Hexane:iPrOH = 99:1
```


DAD1 B, Sig=254,16 Ref=360,100 (E:\GROUP\EXPERIMENT\ASSYMMETRIC ALLYLIC C-H ACTIVATION\HPLC\LQ122300.D)

```
=====================================================================
                       Area Percent Report
=====================================================================

Sorted By            :       Signal
Multiplier           :       1.0000
Dilution             :       1.0000
Sample Amount        :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area       Height    Area
  #   [min]        [min]   [mAU*s]      [mAU]       %
----|-------|----|-------|----------|----------|--------|
  1  12.029 BB    0.3918 1.22926e4    488.12122  97.0157
  2  13.474 BV    0.3326 378.12607    13.84810    2.9843

Totals :                  1.26707e4    501.96931
```
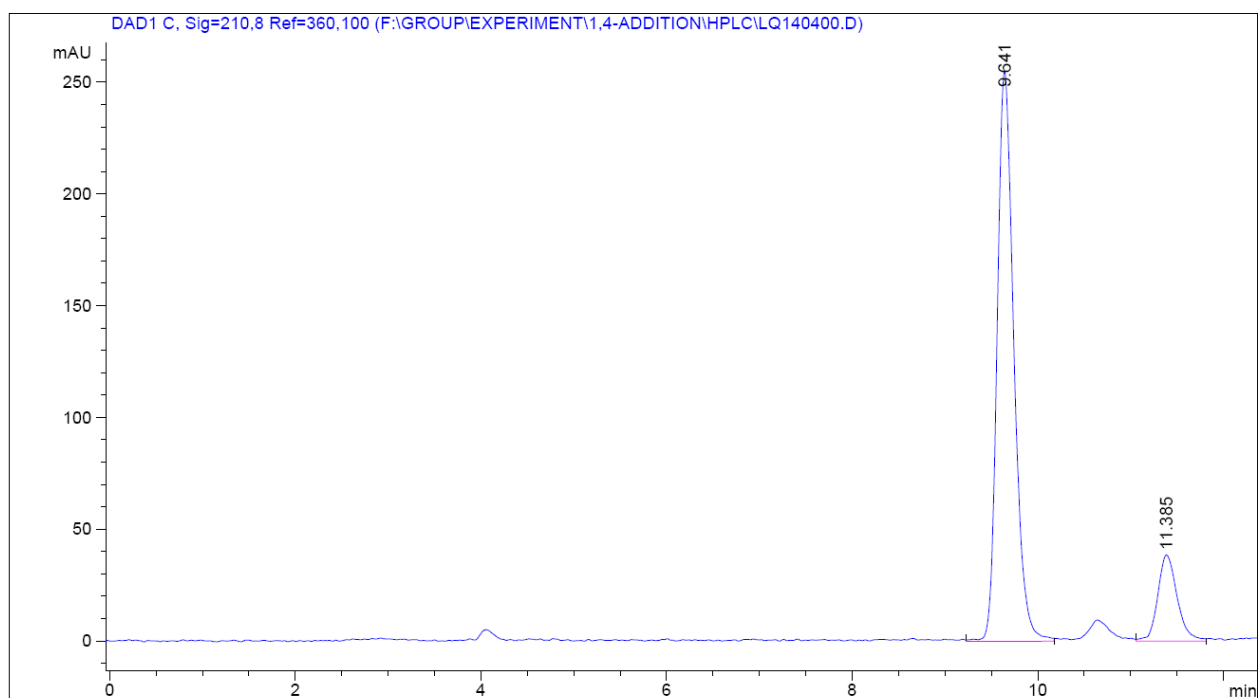
(2) ee of the product

```
Acq. Operator   : LQ
Acq. Instrument : Instrument 1                  Location :   -
Injection Date  : 2010-10-14 4:12:09
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2010-10-14 3:26:10  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : AD-H, 1.0 ml/min, Hexane:iPrOH = 97:3
```


DAD1 C, Sig=210,8 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\LQ132100.D)

```
=====================================================================
                      Area Percent Report
=====================================================================

Sorted By           :       Signal
Multiplier          :       1.0000
Dilution            :       1.0000
Sample Amount       :       1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
  #   [min]        [min]   [mAU*s]      [mAU]        %
----|-------|----|-------|----------|----------|--------|
  1   8.203 VV    0.1680 4362.21191  403.93021  95.3460
  2   9.588 VV    0.1891  212.92461   16.44382   4.6540

Totals :                 4575.13652  420.37403
```
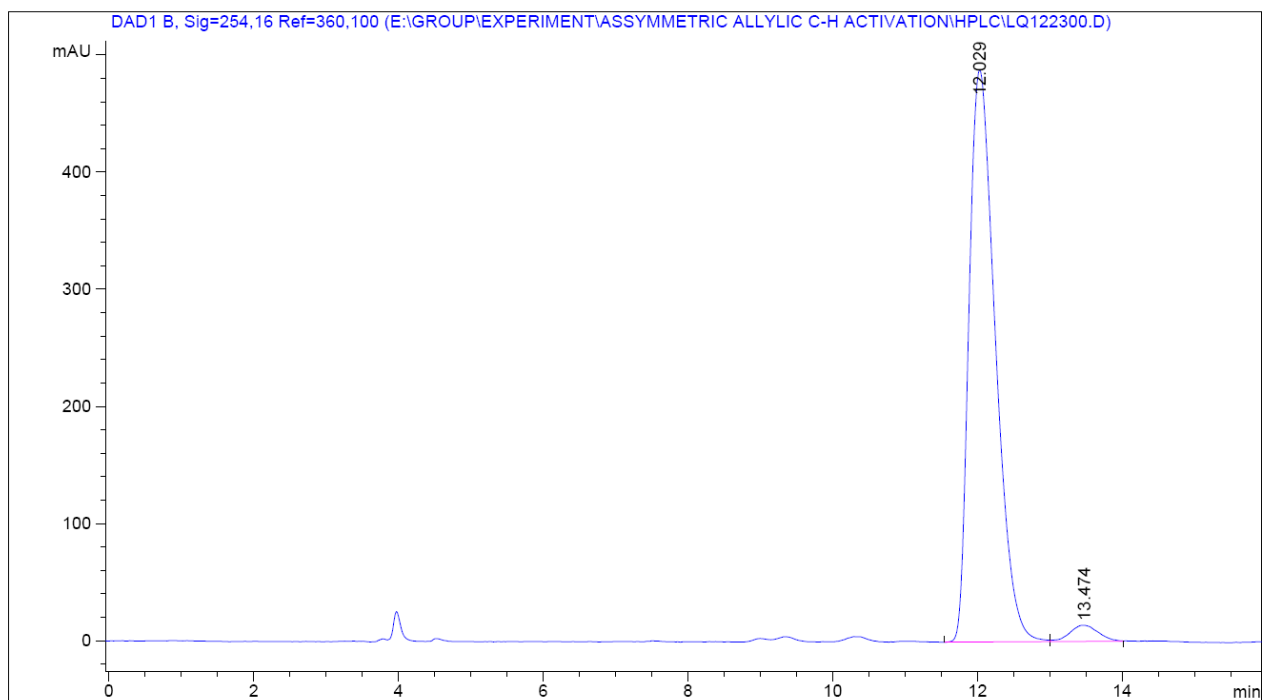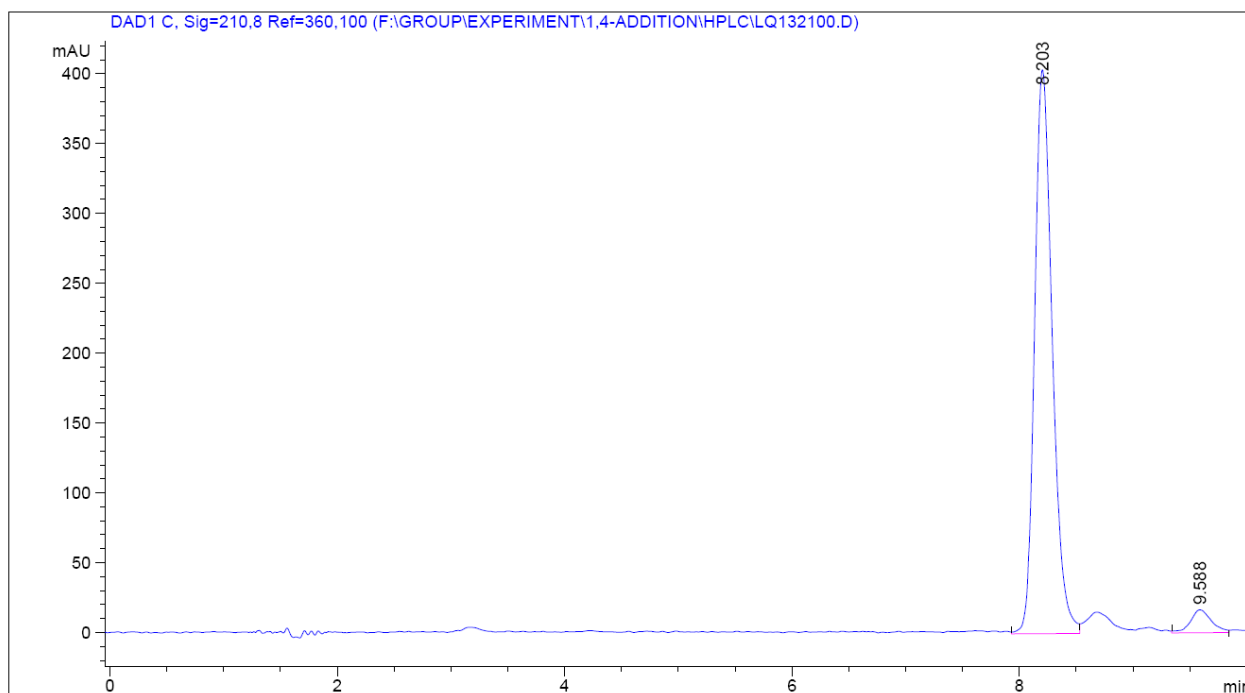
8. The eighth spot

(1) ee of the ligand

Acq. Operator   : LQ
Acq. Instrument : Instrument 1   Location :   -
Injection Date : 2011-1-19 5:40:16
Acq. Method   : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed   : 2011-1-19 4:48:05   by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed   : 2004-4-7 0:10:12
Sample Info   : OD-H, 1.0 ml/min, Hexane:iPrOH = 99:1

```
========================================================================
                        Area Percent Report
========================================================================

Sorted By            :       Signal
Multiplier           :       1.0000
Dilution             :       1.0000
Sample Amount        :       1.00000  [ng/ul]    (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area       Height     Area
  #   [min]        [min]   [mAU*s]      [mAU]        %
----|-------|----|-------|----------|----------|--------|
  1  11.852 VV    0.3348 3636.73730  166.52055 100.0000

Totals :                  3636.73730  166.52055
```
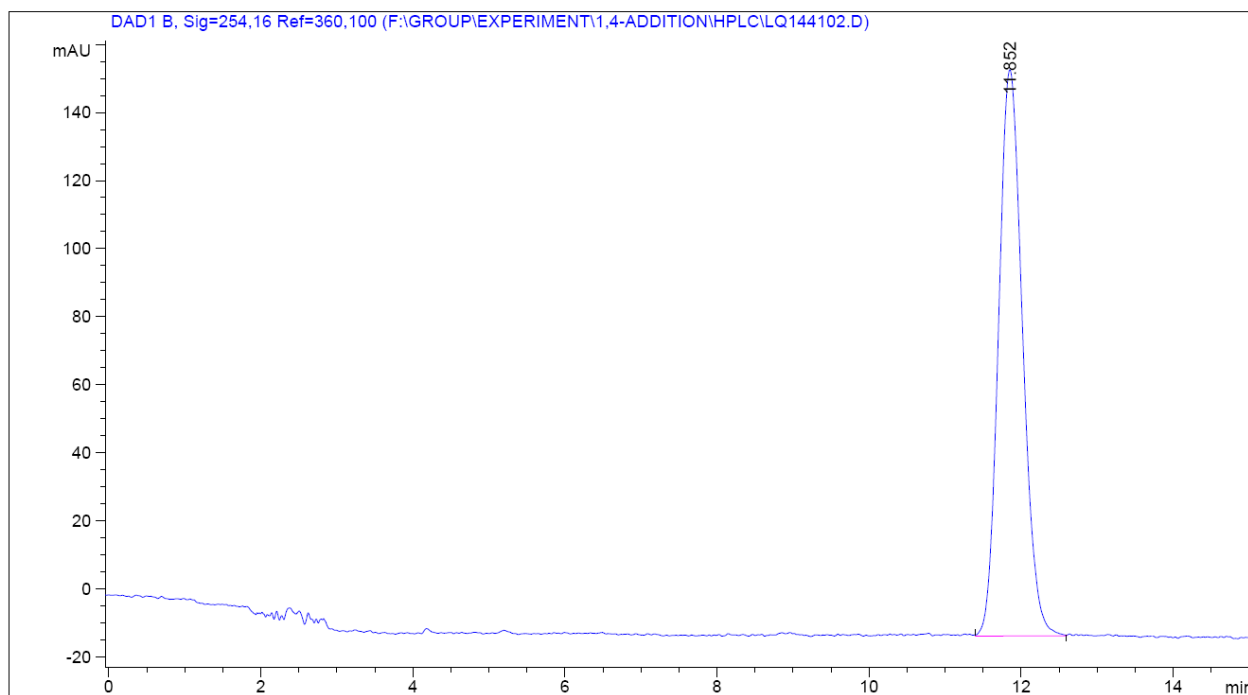
(2) ee of the product

```
Acq. Operator   : LQ
Acq. Instrument : Instrument 1                    Location :   -
Injection Date  : 2011-1-19 11:15:38
Acq. Method     : C:\HPCHEM\1\METHODS\DEF_LC.M
Last changed    : 2011-1-19 9:58:09  by LQ
                  (modified after loading)
Analysis Method : C:\Chem32\1\METHODS\DEF_LC.M
Last changed    : 2004-4-7 0:10:12
Sample Info     : AD-H, 1.0 ml/min, Hexane:iPrOH = 97:3
```


DAD1 C, Sig=210,8 Ref=360,100 (F:\GROUP\EXPERIMENT\1,4-ADDITION\HPLC\LQ148001.D)
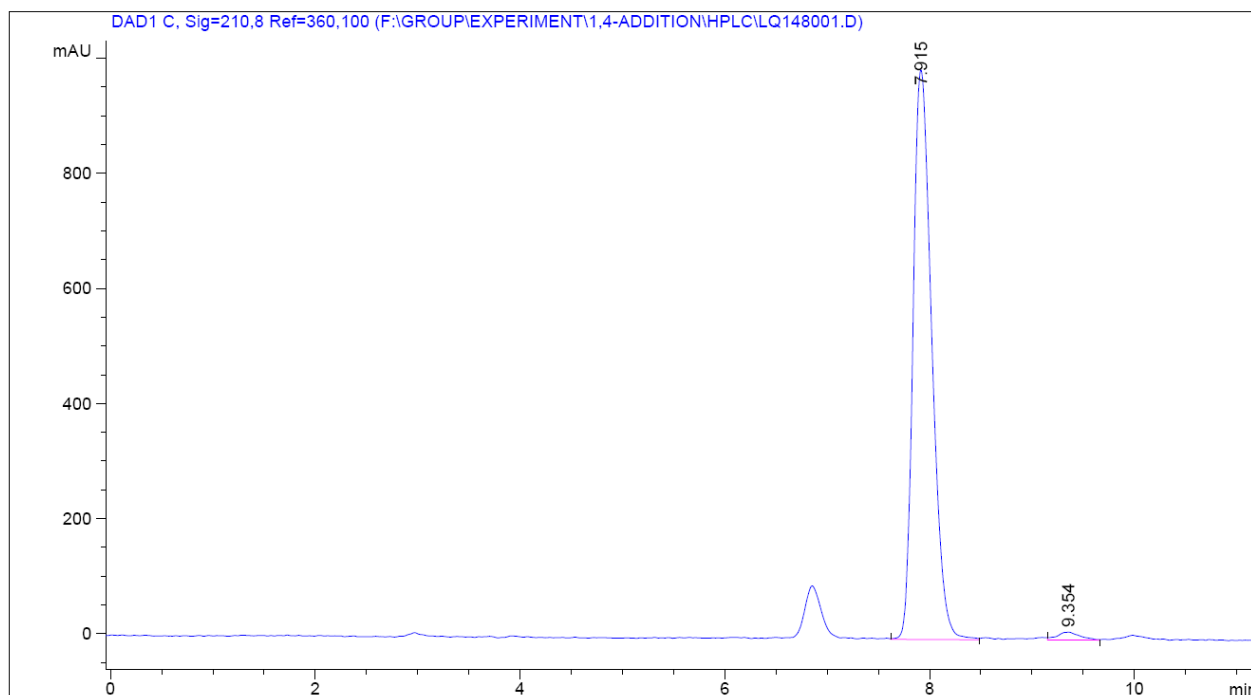
```
=================================================================
                      Area Percent Report
=================================================================

Sorted By          :        Signal
Multiplier         :        1.0000
Dilution           :        1.0000
Sample Amount      :        1.00000  [ng/ul]   (not used in calc.)
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width      Area        Height      Area
  #   [min]        [min]    [mAU*s]        [mAU]        %
----|-------|----|-------|----------|----------|--------|
  1   7.915 VV    0.1912 1.24968e4   990.60791   98.2964
  2   9.354 VV    0.1979  216.58832   13.74019    1.7036

Totals :                  1.27134e4  1004.34810
```